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UDC 543.05

Rapid Low-Temperature Fusion of Specimens by Ultrasound

907M0289A Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 6, Jun 90 (manuscript received 6 Jul 89) pp 1092-1095

[Article by L. I. Pleskach, Ye. P. Yezhova, and G. D. Chirkova, Kazakh State University imeni S. M. Kirov, Alma-Ata]

[Abstract] The purpose of this work was to develop an express method for ultrasonic alkaline fusion of rock and minerals for subsequent atomic absorption determination of their components. It was found that low-frequency ultrasound completely fused the specimens at 200°C in 1.5 minutes and at 240°C in 1 minute. Water or acid leaching of the fused specimens with ultrasonic treatment allows dissolution of the melt in dilute HCl in 1 minute rather than overnight. A specimen size of 1-2 g is sufficient for the determination of most components, although noble metal ores may require a charge of 10-50 g. Figure 1; References 6 (Russian).

UDC 541.49:546.05:547.898

Sorption of Europium by Polymer Sorbent Containing Diaza-18-crown-6 and Its Analytic Application

907M0289B Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 6, Jun 90 (manuscript received 6 Jul 89) pp 1096-1100

[Article by S. V. Beltyukova, T. B. Kravchenko, A. Kh. Zitsmanis, G. M. Balamtsarashvili, A. S. Roska, and Ye. V. Malinka, Institute of Physics and Chemistry imeni A. V. Bogatskiy, UkSSR Academy of Sciences, Odessa]

[Abstract] A study is made of the sorption of tenoyl trifluoroacetonate by europium on a polymer sorbent containing functional groups of the macrocyclic polyester diaza-18-crown-6, and the possibility is demonstrated of using the luminescent properties of the europium on the sorbent for its determination in oxides that damp luminescence. The sorption capacity of the sorbent and the degree of extraction are determined for all the lanthanides. Luminescent determination of europium in oxides of thulium and ytterbium is reported. Figures 2; References 12: 11 Russian, 1 Western

UDC 543:546.46:535.24

Photometric Determination of Magnesium in Air by Reaction With Congo Red and Salicylic Acid

907M0289C Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 6, Jun 90 (manuscript received 1 Jun 89) pp 1173-1177

[Article by O. N. Putilina and V. V. Makarevskaya, Scientific Research Institute of Labor Hygiene and Occupational Diseases, Donetsk]

[Abstract] Conditions for determining magnesium by its reaction with Congo red and various complex-forming substances are investigated, and a photometric method is developed for determining magnesium in air. The method allows for a determination of 0.5-10 µg of magnesium in the presence of calcium, aluminum, and iron with a relative standard deviation of 0.03-0.12. Figures 2; References 7 (Russian).

UDC 543.25.546.22

Ionometric Determination of Potassium in Rock

907M0289D Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 6, Jun 90 (manuscript received 16 May 89) pp 1193-1196

[Article by G. I. Bebshko, All-Union Scientific Research Institute of Mineral Raw Materials, Moscow]

[Abstract] A reliable and highly accurate method is developed for determining potassium in rock. The influence of sulfate, chloride, nitrate, chlorate, acetate, butyrate, succinate, adipinate and trichloroacetate on the functioning of a potassium-selective electrode with a membrane containing valinomicine is studied. The use of an electrode filled with a 0.01 M solution of KCl and a buffer solution containing an organic acid help to increase the accuracy of the determination of potassium in rock. Figure 1; References 8: 7 Russian, 1 Western.

UDC 543.422.28:538.082.74:621.318.14

Phase Diagnosis of Yttrium-Barium Superconducting Ceramic by EPR Spectrometry

907M0289E Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 6, Jun 90 (manuscript received 24 Apr 89) pp 1205-1212

[Article by V. Yu. Nad, T. A. Orlova, L. A. Matveyeva, S. S. Grazhulene, and O. M. Petrukhin, Institute of Microelectronic Technology Problems and High-Purity Materials, USSR Academy of Sciences, Chernogolovka]

[Abstract] A study is presented of the possibility of microwave spectrometry, including EPR, as a method of phase analysis of a yttrium-barium superconducting ceramic. The absorption of microwave radiation by the ceramic specimens in a low-intensity magnetic field can be used for rapid and highly reproducible determination of the superconducting phase. The resolution of the EPR signal in the g=2 area into components corresponding to individual compounds yields information on the content of certain nonsuperconducting phases. Figures 5; References 17: 6 Russian, 11 Western.

UDC 543.423

Chemical-Atomic-Emission Determination of Trace Elements in Biological Fluids

907M0289F Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 6, Jun 90 (manuscript received 6 Jul 89) pp 1234-1237

[Article by G. P. Troshkova and I. G. Yudelevich, Institute of Inorganic Chemistry, Siberian Department, USSR Academy of Sciences, Novosibirsk]

[Abstract] A method is developed for chemical-atomic emission determination of trace elements in blood serum and whole blood by using group concentration of Bi, Co, Cr, In, Mn, Mo, Ni, Nb, Pb, Ti, and V on the chelate-forming sorbent polyarsenazo, which was previously used to determine elements in carbonaceous rock. The limit of detection is $0.8-5.0~\mu g/l$, the relative standard deviation bing 0.15-0.23. Figures 2; References 6: 5 Russian, 1 Western.

UDC 541.183

Determination of Separation Coefficients in Mixtures of Organometallic Compounds by Dynamic Method

907M0315B Moscow VYSOKOCHISTYYE VESHCHESTVA in Russian No 4, Jul-Aug 90 (manuscript received 23 Mar 90) pp 71-73

[Article by S. A. Borisov, N. N. Kuritsyn, O. Ye. Zhuravlev, I. B. Trubnikov, and V. A. Fedorov, Institute of General and Inorganic Chemistry imenin N. S. Kurnakov, Moscow]

[Abstract] A method is presented for calculating the adsorption separation factors in a broad area of compositions of organometallic compounds based on the results of dynamic experiments. Considerable attention is given to the development of an installation to study the dynamics of adsorption of corrosive and highly toxic substances at high temperatures. The experimental installation and method developed were used to study adsorption of a mixture of triethoxyarsine + tetraethoxysilane on carbon. Satisfactory agreement is determined between the static and dynamic methods. Figures 2; References 4 (Russian).

UDC 543.312:546:543.253

Some Features of Water Analysis

917M0004A Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 8, Aug 90 (manuscript received 12 Dec 88) pp 1608-1614

[Article by V. F. Osyka, L. Ya. Kheyfets, A. V. Cherevik, and S. G. Maksimovskiy, All-Union Scientific Research Institute of Water Protection, Kharkov]

[Abstract] The complexity of water analysis is due mainly to (1) the continuous variability of the substances being determined and the continuous variability of the system as a whole owing to its simultaneously occurring physical, chemical, and biological processes and (2) the possibility that the forms in which analyzed substances exist in water may differ greatly from the standpoint of toxicity and bioaccessibility, migration capability, and participation in the biogeochemical cycle. Water analysis is further complicated by the existing standardization system, which requires determining a number of individual substances in minute concentrations in the presence of far greater concentrations of different impurities without precise specification of standardized forms or any correspondence between the specified forms and the state of substances in water. By way of the example of the polarographic determination of four individual metals, i.e., vanadium (V), chromium (VI), cadmium, and zinc, the authors of this article present practical recommendations regarding limiting the changes in the state of substances in water that are undergoing analysis. Techniques for preparing samples for analysis and for conducting the actual analysis are outlined. It is concluded that the minimum effect on the state of the substances being analyzed is achieved by using difference methods, particularly by using differential chronoamperometry and using a portion of the analyzed water as a reference solution. Figure 1, tables 2; references 6 (Russian).

UDC 543.38:541.14:404.4.064.3

Simultaneous Determination of Dissolved Organic Forms of Carbon and Phosphorus in Natural Waters With Photochemical Oxidation of Organic Matter by Using Low-Pressure Mercury Lamps

917M0004B Moscow ZHURNAL ANALITICHESKOY KHIMH in Russian Vol 45 No 8, Aug 90 (manuscript received 5 Apr 89) pp 1615-1623

[Article by A. N. Shkil, A. V. Krasnushkin, and I. T. Gavrilov, Moscow State University imeni M. V. Lomonosov]

[Abstract] The existing method of determining the total content of dissolved forms of nitrogen and phosphorus in natural waters by mineralizing the dissolved organic matter contained in those waters is inefficient and complicated by a number of factors. The medium- and high-pressure mercury-quartz lamps that have been used in the procedure for about 12 years have a low fraction of UV radiation in their total radiation spectrum but high

fractions of visible and IR radiations. This causes the irradiated solution to boil, which in turn necessitates increasing the distance between the lamp and the irradiated solution and thus sharply reduces the efficiency of the photomineralization process and increases the complexity of the equipment used to implement it. To date. only one study has considered the feasibility of switching over to low-pressure lamps. The study reported herein examined the possibility of increasing the efficiency of the process by using a low-pressure mercury lamp to effect the photochemical oxidation of dissolved organic matter to make a simultaneous determination of the dissolved organic carbon and total amounts of phosphorus in natural waters. The method developed is based on photomineralization of dissolved organic matter, coulometric determination of evolved carbon dioxide. and spectrophotometric determination of orthophosphate in the form of molybdenum blue. The method's accuracy was tested by using the method of additives with four samples of natural waters containing a total content of 30 to 50 µg/l phosphorus. The mean error of 4-5 measurements ranged from 3 to 5 µg/l with a relative standard deviation from 0.04 to 0.12% (P = .95). The method's accuracy was also found to be satisfactory from the standpoint of determination of dissolved organic carbon in the samples analyzed. Figures 4, tables 3; references 14: 6 Russian, 8 Western.

UDC 546.426:546.54

Determining Iron in Waters by Solid-Phase Spectrophotometry With Membrane Filtration

917M0004C Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 8, Aug 90 (manuscript received 18 Jul 89) pp 1624-1628

[Article by A. T. Pilipenko, A. V. Terletskaya, and T. A. Bogoslovskaya, Colloidal Chemistry and Water Chemistry Institute imeni A. V. Dumanskiy, UkSSR Academy of Sciences, Kiev]

[Abstract] Solid-phase spectrophotometry has definite advantages over other methods of analyzing a concentrate in that it rationally combines the sorption concentration of elements from a solution and their determination based on measurement of the sorbent's light-absorbing phase. Using solid-phase spectrophotometry with membrane filtration makes it possible to reduce the detection limit of the elements being determined. The main difficulties encountered when the method is used are related to obtaining a light-absorbing layer that is evenly filled with ion exchanger and measuring its light absorption. The authors of this article developed a variation of the solid-phase spectrophotometry method with membrane filtration. The method entails production of a dyed iron compound, its sorption by a finely

dispersed ion exchanger, separation on a membrane filter, and direct measurement of the concentrate's light absorption (or diffuse reflection). This method of producing a dyed layer that is suitable for photometry on a membrane filter was found to greatly simplify the analvsis process. The method was found to have the following detection limits (µg/1): 0.2 for bathophenanthroline, 0.5 for 1,10-phenanthroline, and 0.8 for 2,2'dipyridyl. Combining trace iron sorption by a KU-23 macroporous highly dispersed cation exchanger with separation of the concentrate on a membrane filter and measurement of the coloration intensity of the thin layer on the filter made it possible to reduce the detection limit. The method of determining iron with 1,10phenanthroline was used to control the quality of deionized water and to determine different forms of iron in low-mineralized natural waters. Figures 3, tables 3; references 11: 7 Russian, 4 Western.

UDC 543.544:543.4

Determining Benzoic and Sorbic Acids and Their Salts in Foodstuffs

917M0004D Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 8, Aug 90 (manuscript received 18 Apr 89) pp 1640-1646

[Article by D. B. Melamed, B. G. Lyapkov, and F. A. Medvedev, Nutrition Institute, USSR Academy of Medical Sciences, Moscowl

[Abstract] High-performance gas and liquid chromatographic methods have received a great deal of attention as methods for use in determining the presence of benzoic and sorbic acids and their salts in foods and beverages. However, both methods require expensive and complex equipment. Thin-layer chromatography (TLC) is a simpler way of isolating benzoic and sorbic acids, separating them from the impurities in the final extract, and ultimately identifying them. Furthermore, TLC makes it possible to implement the reactions confirming the presence of the compounds being determined on the chromatographic plate. The goal of the authors of this article was to perfect the methods of determining benzoic and sorbic acids and their salts in foods and beverages by increasing the degree of separation in a thin silica gel layer and creating a new method of confirming the correctness of their identification. The analysis method they developed combined the use of high-performance gas chromatography and spectrophotometry. The method's detection limits are 5 µg for benzoic acid and 50 ng for sorbic acid with a standard deviation not exceeding 0.18. A comparison of the results achieved by using the new method and those achieved by high-performance liquid chromatography indicated that the new method is reliable and has satisfactory reproducibility. Figure 1, tables 3; references 14: 7 Russian, 7 Western.

UDC 669(546.74+546.621)

Structure of Raney Nickel Catalyst Obtained From Mechanically Alloyed Nickel Aluminides

907M0296A Novosibirsk IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKIKH NAUK in Russian No 3, May 90 (manuscript received 16 Jun 89) pp 60-62

[Article by G. V. Golubkova, Ye. Yu. Ivanov, and T. F. Grigoreva, Institute of Solid-State Chemistry and Processing of Mineral Raw Materials, Siberian Department, USSR Academy of Sciences, Novosibirsk]

[Abstract] It has been found possible to produce nickel aluminides by the new method of mechanical alloying and processing of powders of the initial components in various types of act, vators. The area of existence of NiAl with a CsCl structure is extended by this method from 33 to 68 at% Al. Nickel aluminide thus produced contains over 50 at% aluminum and has both nonstoichiometric defects resulting from the formation of a solid solution as well as structural defects of plastic deformation resulting from the mechanochemical synthesis. Heating of specimens to over 200°C causes annealing of the defects with liberation of heat due to the energy stored in the mechanical alloying process, the amount of which increases with increasing variation from the stochiometric composition. Treatment in 20% KOH in a boiling water bath for 2 hours resulted in removal of aluminum nickel aluminide powders of all compositions studied. X-ray phase analysis reveals a mixture of cubic nickel and CsCl-type NiAl. The retention of the CsCl-type structure indicates that processes of recrystallization or breakdown of the initial lattice practically do not occur during leaching. thus indicating the anomalous mobility of the aluminum atoms in the initial NiAl lattice due to the high defect content of the specimens, which form vacancy clusters in the highly deformed metals. Figure 1; References 9: 6 Russian, 3 Western.

UDC 542.973.2

Influence of Antimony Compounds on Formation of Iron-Antimony Oxide Catalyst

907M0296B Novosibirsk IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKIKH NAUK in Russian No 3, May 90 (manuscript received 15 Nov 89) pp 63-66

[Article by G. A. Zenkovets, and D. V. Tarasova, Institute of Catalysis, Siberian Department, USSR Academy of Sciences, Novosibirsk]

[Abstract] A study is presented of the influence of an antimony compound on the formation of the phase composition and catalytic properties of an iron-antimony oxide catalyst produced by mixing. The catalysts were obtained by adding a solution of iron nitrate to a gel of antimony oxide with agitation and then aging at 80°C for 3 hours with intense agitation followed by

drying and heat treatment. The phase composition of the specimens was studied by x-ray phase analysis in filtered CuKa radiation. The results indicate that when the solution of iron nitrate contacts compounds obtained by the oxidation of antimony trioxide, in all cases during subsequent drying the iron ions react with the antimony oxides to form products, the composition and structure of which are determined by the nature of the initial antimony oxide. The end products of heat treatment are always iron antimonate and the oxide α-Sb₂O₄. Formation of the phase composition of the iron-antimony specimens occurs variously, depending on the initial antimony compound. Regardless of the initial antimony compound, catalysts heated to 500°C achieve a high rate of propylene conversion and good selectivity for acrylonitrile. Good effectiveness of iron-antimony catalysts is achieved by the absence of unbonded iron oxide. Completeness of bonding of the components is achieved by the interaction of iron ions with the antimony oxides in the early stages of synthesis, during mixing and drving. References 9: 8 Russian, 1 Western.

UDC 54.057.542.943.7:54-36.546.73.546. '11.546.96

Synthesis of Immobilized Ru, Co, and Mn Hydroxides and Catalytic Properties in Oxidation of Water

907M0296C Novosibirsk IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKIKH NAUK in Russian No 3, May 90 (manuscript received 14 Nov 89) pp 86-93

[Article by G. L. Yelizarova, L. G. Matviyenko, N. V. Lozhkina, V. N. Parmon, and E. M. Moroz, Institute of Catalysis, Siberian Division, USSR Academy of Sciences, Novosibirsk]

[Abstract] A unified approach is taken to the synthesis of water-oxidation catalysts for various ions. The method is based on the hydrolysis of salts or complexes of Co, Mn, or Ru on the surface of traditional carriers such as zeolites, TiO₂, SiO₂, and Al₂O₃; highly active and reproducible specimens are produced. This forms highly dispersed hydroxides on the carrier surfaces that are fragmented by the anions in the reaction mixture. Figure 1; References 14: 5 Russian, 9 Western.

UDC 661.183.6+541.128.13+549.67

Organic Synthesis on Zeolite Catalysts

907M0296D Novosibirsk IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKIKH NAUK in Russian No 3, May 90 (manuscript received 1 Nov 89) pp 103-119

[Article by A. G. Ione, Institute of Catalysis, Siberian Division, USSR Academy of Sciences, Novosibirsk]

[Abstract] Crystalline zeolites in decationized form are acid-base catalysts that accelerate reactions in which

traditional catalysts are frequently used, including electrophilic substitution in the aromatic ring, alkylation, nitrogenation, and chlorination of aromatic and cyclic compounds. Studies of the selectivity of electrophilic substitution on zeolite catalysts have shown that the isomer composition of the substitution products depends on the reaction conditions and type of zeolite. Disproportionation and dialkylation may also occur, as when mineral acids and metal halides are used. The use of zeolite catalysts can allow the creation of processes based on heterogeneous catalytic systems, in many cases avoiding the use of mineral acids and Friedel-Crafts reagent, thus improving economic results and reducing pollution. References 134: 10 Russian, 124 Western.

UDC 541.128.13+543.422.4+547.551.2

Electrophilic Substitution on Zeolite Catalysts. 2. Mechanism of Alkylation of Aniline by Methanol

907M0296E Novosibirsk IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKIKH NAUK in Russian No 3, May 90 (manuscript received 1 Nov 89) pp 125-130

[Article by L. V. Malysheva, O. V. Kikhtanin, Ye. A. Paukshtis, and K. G. Ione, Institute of Catalysis, Siberian Department, USSR Academy of Sciences, Novosibirsk]

[Abstract] IR spectroscopy is used to study the nature of active centers and the form of interaction of reagents with the surface of the catalyst in the alkylation of aniline by methanol on H-ZSM-5 zeolites modified by compounds of phosphorus and magnesium, as well as AlPO-5, AIPO-11, and AIPO-25 at 25-400°C. The data obtained confirm the assumption that the basic features of the electrophilic substitution reaction remain the same when it occurs on the surface of heterogeneous catalysts: the protonated aniline is practically not alkylated on the aromatic ring, and the molecular form of the aniline is alkylated by surface methoxy groups to form ortho- and para-isomers. The zeolite catalysts distribute the acid centers over the surface within the channels or on the external surface of the crystals. Rules for regulating the selectivity of the alkylation reaction are presented. Figures 5; References 7: 5 Russian, 2 Western.

UDC 532.51:66.097.3

Optimal Dimensions for Bulk Catalyst

917M0005A Moscow ZHURNAL PRIKLADNOY KHIMII in Russian Vol 63 No 8, Aug 90 (manuscript received 10 Jul 89) pp 1658-1662

[Article by A. V. Bespalov, Ye. Yu. Shinkovskaya, V. S. Beskov, and Yu. A. Volynkin, Moscow Chemical Technological Institute imeni D. I. Mendeleyev] [Abstract] When selecting the optimal shape and dimensions of a catalyst's grains one must consider that the degree to which the inner surface is utilized and the

layer's pressure loss decrease as the grain size increases. Other constraints on the maximum grain size arise if the catalytic process becomes less selective as the grain size increases, and the shape of the catalyst grains affect the degree of the catalyst layer's structural heterogeneity. It is in the context of these factors that great interest has been expressed in catalysts with new geometric shapes, specifically block catalysts with a "honeycomb structure." The study reported here shows that the speed of the catalytic reaction in relative units (W_{rel}) is naturally referenced to the speed of the reaction on honeycomb grains in a bulk grain layer in the kinetic range. The authors conclude that the maximum of this ratio will correspond to the maximum of the degree to which a honeycomb grain with large channels (and possibly, with thinner layers between them) is used. After making a series of calculations and conducting a series of experiments, they conclude that the inner surface of block channels that are positioned at an angle of 90° relative to the oncoming flow is accessible and that there exists an optimal radius size for through channels that should be selected with an allowance for the circulation of the block and on the degree to which it is utilized. They further conclude that the final selection of a catalyst block's thickness should be determined by the specific chemical reaction being conducted and the strength properties and technological feasibility of the block. Figures 3; references 9 (Russian).

UDC 541.128

Effect of Technological Performance Parameters on Properties of Bulk Silver Catalyst for Formaldehyde Synthesis

917M0005B Moscow ZHURNAL PRIKLADNOY KHIMH in Russian Vol 63 No 8, Aug 90 (manuscript received 7 Dec 89) pp 1853-1854

[Article by A. A. Sakharov, A. N. Pestryakov, N. V.

Sakharova, and L. N. Kurina] [Abstract] Silver catalysts are used extensively in the large-scale commercial process of oxidizing methanol into formaldehyde. In the USSR more than 90% of formalin is produced on an applied silver catalyst. In view of the fact that the recent trend in domestic formalin lines is to use a bulk silver catalyst, it has become critical that the operating modes of bulk silver contacts be studied and that ways of preparing them be perfected. To improve the process of synthesizing formaldehyde, the authors of the study reported here developed a new method of preparing SD-1 bulk silver catalyst and studied the effect that the parameters of the process in which it is used have on its catalytic properties. The tests reported were conducted on a flowthrough catalysis unit with close-to-commercial process parameters: reactor temperature, 500-600°; an O₂:CH₃OH ratio of 0.18 to 0.33; concentration of methanol in the water-and-alcohol solution, 70-100%, methanol load on the reactor, 80 g/(cm²·h); and height of the catalyst layer, 1.5 cm. The process was implemented in

an adiabatic mode at 500°, whereas additional heating of the reactor was used at 550 and 600°. The gaseous reaction process was subjected to chromatography, and the condensed products (CH₂O and HCOOH) were analyzed by titration. The CD-1 bulk silver catalyst was found to work effectively in both "soft" and "hard" regimens, with the optimal temperature being 500-550° (which is achieved in a "soft" regimen). Increasing the O₂:CH₃OH ratio raised the yield of formaldehyde; however, because temperature is strictly correlated with the component ratio in commercial adiabatic reactors, increasing the O₂:CH₃OH ratio in such reactors

increases the temperature, thereby resulting in additional thermal breakdown of the formaldehyde and reducing the selectivity of the process. It was also discovered that using 100% methanol is best in the "soft" regimen, although a "semihard" regimen in which methanol is diluted by water to 90-95% may in some cases be preferable in order to boost the formaldehyde yield. Finally, it was determined that including a product rectification cycle in the formaldehyde line and returning unreacted methanol to the reaction cycle reduced the methanol consumption coefficient. Table 1; references 3 (Russian).

UDC 338.5 54+483 620 2

Relationship of Purity and Price of Substances

90. MO315N Moscow VYSOKOCHISTYYF VESHCHESTVA in Russian No 4, Jul. Aug 90 imanuscript received 30 Oct 89) pp 220-233

[Article by L. A. Niselson, and O. N. Kalashnik, State Scientific Research and Planning Institute for the Rare Metals Industry Giredmet, Moscow]

[Abstract] An analysis is presented of the relationship between purity and the price of materials. The author states that in a free market and natural economy the cost represents most of the price of a product and can therefore be considered proportional. Since cost data are protected as commercial secrets, the analysis here must be based on price. Extensive tables compare the state standard prices and prices computed from equations based on cost for a wide variety of materials. The price of a material is an important characteristic that is related to many factors. One of the principle factors that determine the price of high-purity materials is their purity. Price, however, varies with the level of consumption and the development of technology for the production of highly pure substances. However, the observed relationship between price and purity is thought to be one of the more stable factors involved in price determination. References 15: 14 Russian, 1 Western.

UDC 536.462:541.182.024

Numerical Estimates of Flame Temperature During Synthesis of Highly Dispersed Silicon Oxide

917M0005C Moscow ZHURNAL PRIKLADNOY KHIMII in Russian Vol 63 No 8, Aug 90 (manuscript received 18 July 89) pp 1701-1705

[Article by O. V. Kravchuk and A. A. Chuyko, Institute of Surface Chemistry, UkSSR Academy of Sciences] [Abstract] The authors of the study reported here derived numerical estimates of the flame temperature of the reaction of the formation of pyrogenic silicon oxide. Unlike the conventional method of calculating the temperature of an adiabatic flame, which includes a procedure for making the system of conservation and equilibrium equations self-consistent, the procedure developed here was based on the hypothesis that up until some temperature the gaseous mixture consists of reagents and that after that temperature it consists of products. This

assumption greatly simplified the calculations required, and comparative calculations showed a good coincidence of results at the reagent-to-product transition temperature of 800°. The authors established that the calculated flame temperature is proportional to the initial temperature of the gaseous mixture and is described by the equation $T = A + 0.7425T_0$ (where A is a numerical coefficient, i.e., $A = 1.801^{\circ}$ for a stoichiometric mixture with air as the oxidizing agent) and T₀ is the initial temperature of the mixture. The authors concluded that, depending on the temperature and length of the high-temperature zone of the flame jet, there is a cessation of particle growth due to both a decrease in temperature and an increase in particle size. They thus determined that changing the concentration of reagents in the reaction mixture and the flame jet temperature makes it possible to obtain a product not only with different degrees of dispersion but also with different particle size distributions. Figures 4; references 7 (Russian).

UDC 546.491

C. rbon-Containing Wastes From Swirl Burning of Coal at TETs as Adsorbents of Mercury Anions From Dechlorinated Liquors

917M0005D Moscow ZHURNAL PRIKLADNO' KHIMII in Russian Vol 63 No. 8, Aug. 90 (manuscript received 14 Apr. 89) pp. 1684-1688

[Article by R. P. Kochetkova, E. I. Sorkin, I. P. Shiverskaya, V. V. Voronkov, V. L. Apasov, and V. V. Polyakov, Petrochemical and Coal Chemical Synthesis Institute, Irkutsk State University]

[Abstract] In the commercial practice of removing mercurv from the dechlorinated depleted mother liquor that results from the production of chlorine and alkali by electrolysis with a mercury cathode, sorption extraction methods are commonly used together with methods of precipitating mercury in the form of sulfides. Large-pore activated charcoal with anion-exchange properties has been recommended for extracting mercury from mother liquor. Its high cost and scarcity prevent this practice from being adopted, however. As a way of circumventing this problem, the authors of the study reported here proposed using the carbon-containing wastes from the swirl burning of coal at TETs as an unconventional and inexpensive sorbent for extracting mercury ions. During the course of their research, the authors examined seven different specimens of coke crumb, including three specimens activated to different degrees, an oxidized specimen, and a specimen that had been oxidized and heat-treated in a nitrogen current. It was found that the coke crumbs produced during swirl burning have anionexchange properties. It was further discovered that, after boiling-layer activation by a steam and gas mixture consisting of steam and carbon dioxide, coke crumb may be used as a carbon adsorbent to extract mercury anions from the mother liquor. The chemical nature of the surface of the coke crumbs was found to be what largely determines the depth of the sorption of mercury anions from the liquor. When the surface of the initial coke crumb is activated by a gas mixture, its anion-exchange properties increase; they reach their minimum during oxidation. Temperature treatment of oxidized coke crumbs in a flow of inert gas restores the coke crumbs' anion-exchange properties. Figures 4, table 1; references 10: 9 Russian, 1 Western.

UDC 541 183 03:33.2 547 551 1.547.562.1

Processes of Ion-Exchanger Sorption of Organic Matter From Water

917M0005E Moscow ZHURNAL PRIKLADNOY KHIMII in Russian Vol 63 No 8, Aug 90 (manuscript received 25 May 89) pp 1838-1840

[Article by T. V. Chupina, A. V. Sofronova, and N. I. Volkova, Perm Polytechnic Institute]

[Abstract] Despite the fact that more than 300 types of products require demineralized water, ion exchange remains the sole commercial method of demineralizing water. The efficiency of the ion-exchange process depends on a variety of factors, and actual ion-exchange processes often proceed far from ideally. The authors of the study reported here studied components contained in the waters of the Volga, specifically in the basin of the Kamy River, that affect the ion-exchange process of demineralizing water. For their research, the authors used a highly acidic cation exchanger (KU-2-8) in the H-form and highly basic (AV-17-8) and weakly basic (AN-31) anion exchangers in OH- and Cl-forms that were prepared in accordance with All-Union State Standards [GOST] 20298-74 and 20301-74. Quantities of 5 g ion exchanger were added to flasks containing 200 cm of solutions of phenol and aniline in different concentrations. They were shaken for 10 hours and then thermostatted at a temperature of 20° for 24 hours in a resting state so as to reach a state of equilibrium (which was confirmed by the constancy of the phenol and aniline concentrations in two successive specimens taken after 24-30 hours. The colorimetric method described elsewhere was used to determine the phenol and aniline concentrations. The studies of the isotherms of the sorption of aniline and phenol from their aqueous solutions on the three ion exchangers established that KU-2-8 cation exchanger has a high aniline capacity and that the AN-31 anion exchanger has a high phenol capacity. The ion exchangers' aniline and phenol capacities were found to be a function of the overall content of salts in the solution and decreased as this concentration increased. The anion exchangers' capacities for aniline and phenol in chloride form is lower than in hydroxyl form, Figures 3; references 7 (Russian).

UDC 543.064

High-Purity Solid Substances. Problems of Metrology

907M0315F Moscow VYSOKOCHISTYYE VESHCHESTVA in Russian No 4, Jul-Aug 90 (manuscript received 06 Mar 90) pp 128-134

[Article by G. G. Devyatykh, I. D. Kovalev, S. V. Yankov, T. A. Burmistrova, and L. I. Osipova, Institute of Chemistry of Highly Pure Substances, USSR Academy of Sciences, Gorkiy]

[Abstract] A file of data collected by the National Permanent Exhibit and Collection of Highly Pure Substances that encompasses some 16,000 determinations of elements indicates that in 40% of cases the data of two laboratories analyzing the same specimen diverge by a factor of more than 10. Most of these divergences are systematic errors resulting from the objective difficulty of analysis of highly pure substances. This article discusses factors specific for the metrology of highly pure substances in analytic chemistry and the possibility of using standard specimens for their resolution. The following problems must be solved: development of a technology for manufacture and storage of specimens, development of methods of certification of composition and statistical processing of experimental data, development of industry and state standards defining the process of certification, and development of methods for analvsis of highly pure substances with the use of standard specimens. Figures 2; References 17: 14 Russian, 3 Western.

UDC 543.544

Gas Chromatographic Analysis of the Hydrides of Arsenic, Phosphorus and Their Mixtures With Hydrogen

907M0315G Moscow VYSOKOCHISTYYE VESHCHESTVA in Russian No 4, Jul-Aug 90 (manuscript received 19 Jul 89) pp 145-150

[Article by V. I. Mayorov, L. N. Morozova, and A. D. Molodyk, All-Union Scientific Research Institute of Electronic Technology Materials, Kaluga]

[Abstract] The purpose of this work was to develop a highly sensitive method for monitoring arsenic, phosphorus, and their gas mixtures for their content of gaseous impurities and the content of the hydride in a mixture with hydrogen. A method of gas chromatographic analysis is developed that allows a determination of the impurities N₂, O₂, Ar, CO, CO₂, N₂O, H₂O, CH₄, C₂H₂, C₂H₄, C₂H₆, C₃H₈, n-C₄H₁₀, iso-C₄H₁₀, SiH₄, GeH₄, PH₃ in AsH₃, and AsH₃ in PH₃. The limit of detection of impurities by the heat-conductivity detector with a specimen volume of 0.5 1 is (0.5-1.0):10⁻⁴%. The correctness of the method was tested by performing parallel analysis of specimens by the method and a

gravimetric method. The relative standard deviation was 0.03 at probability 0.95. Figures 4; References 13: 12 Russian, 1 Western.

UDC 543.4

Determination of Tantalum, Titanium, and Chromium Impurities in Niobium Pentoxide by Spectrophotometric Method

907M0315H Moscow VYSOKOCHISTYYE VESHCHESTVA in Russian No 4, Jul-Aug 90 (manuscript received 10 Dec 89) pp 168-170

[Article by S. U. Kreyngold and Ye. M. Yutal, Institute of Electronics Equipment Scientific Production Association, Moscow]

[Abstract] Spectrophotometric methods are described for determining 10⁻³% Ta, 2·10⁻⁴% Ti, and 1·10⁻⁵% Cr in niobium pentoxide and other compounds of niobium. Tantalum is extracted from a solution of 0.4 M HF and 0.05 M H₂SO₄ and then determined by titration with malachite green. Titanium is determined after quadruple extraction by a kinetic method, by the oxidation of o-phenylinediamine by hydrogen peroxide in the presence of an activator and ammonium oxalate. Chromium is determined by the use of diphenyl carbazide in the presence of an excess of fluorides. Figure 1; References 6: 5 Russian, 1 Western.

UDC 621.315.592.546.26

Determination of Carbon in Semiinsulating Gallium Arsenide by Calibrated Optical Method

907M03151 Moscow VYSOKOCHISTYYE VESHCHESTVA in Russian No 4, Jul-Aug 90 (manuscript received 15 Feb 90) pp 171-174

[Article by G. I. Aleksandrova, M. A. Ilin, and N. G. Mikhaylova, State Scientific Research and Planning Institute of Rare Metals Industry, Moscow]

[Abstract] The major methods of determining the concentration of carbon in semiinsulating gallium arsenide are listed and discussed. An attempt is made to determine the value of the calibration coefficient C for use in an optical method of monitoring the concentration of carbon since the error in previous determinations published in the literature has been 10-30%. Systematic calibration of the data of one method by means of the data produced by another method yield a value of $C = (8.8 \text{ plus or minus } 3.0) \cdot 10^{15} \text{ cm}^{-1}$ for optical measurements at a temperature of 80 K. Figure 1; References 14: 5 Russian, 9 Western.

UDC 621.315.592

Small Acceptors and Deep Level Defects in GaAs Layers Doped With Sulfur

907M0315J Moscow VYSOKOCHISTYYE VESHCHESTVA in Russian No 4, Jul-Aug 90 (manuscript received 6 Dec 89) pp 178-180

[Article by T. I. Kolchenko, V. M. Lomako, and Yu. Yu. Fedorov, Scientific Research Institute of Applied Physics Problems imeni A. N. Sevchenko, Minsk]

[Abstract] Methods of low-temperature photoluminescence and nonsteady capacitance deep-level spectroscopy are used to study the influence of sulfur doping on the relative concentration of small acceptors of various type, and concentrations in deep centers of GaAs layers. The layers were grown in the system Ga-AsCl₃-H₂ in an installation with a vertical reactor and flat rotating pedestal. Sulfur doping was performed from a gas mixture of sulfur hexafluoride and helium. It is shown that doping with sulfur up to about 1016 cm-3 causes an increase in the concentration of acceptor zinc impurity with respect to acceptors in the As(Si, C) sublattice, stabilization of the set of point defects, and a decrease in the concentration of E2 and E4 electron traps in the epitaxial layer, which is quite important in practice. Figures 3; References 8: 1 Russian, 7 Western.

UDC 546.284:546.88:54.43

Investigation of Indicator Properties of Vanadium-Modified Silicas and Zeolites

917M0005F Moscow ZHURNAL PRIKLADNOY KHIMII in Russian Vol 63 No 8, Aug 90 (manuscript received 17 Jul 89) pp 1674-1679

[Article by N. G. Belotserkovskaya, D. P. Dobychin, G. M. Kesareva, V. I. Malkiman, V. N. Pak, and V. M. Shamrikov]

[Abstract] Vanadium silica gels are known visual moistures indicators. Their indicator properties result from the fact that vanadium in surface groupings is capable of a reversible coordination complexing of two water molecules that is accompanied by a change in color. The study reported here examined the effect of the nature of the carrier, its pore structure, optical properties (transparency), and content of graft vanadium on the indicator properties of a series of vanadium-containing systems. KSK-1 and N2B large-pore silica gels, glassy silica gels (KSS-4S medium-pore silica gel and a small-pore laboratory specimen), porous glass specimens, and types NaA and NaY zeolite were used as carriers for the vanadium moisture indicators. The tests conducted confirmed the possibility of producing vanadium silica color moisture indicators by processing silica gels and porous glasses with a solution of vanadium chloroxide in carbon tetrachloride. It was discovered that the chemical nature of silica, porous glass, and silica gel and their pore

structure do not affect the indicator properties of vanadium silicas over a rather broad range and that vanadium silicas initiate a relative humidity of 0.1 to 20%. Glassy silica gels or porous glasses were found to be suitable for use as carriers for vanadium indicators. Chalky silica gels and NaA and NaY zeolites, while possible candidates for use as vanadium moisture indicators, are less promising than vanadium silicas because vanadium zeolites are not as bright and, consequently, their range of applicability is narrower. Figure 1, tables 3; references 13: 11 Russian, 2 Western.

UDC 669.74.782.5.046.462:536.421

Thermodynamics of Reduction of Elements' Oxides During Thermal Carbon Process of Smelting Complex Silicon- and Manganese-Based Alloys

917M0005G Moscow ZHURNAL PRIKLADNOY KHIMH in Russian Vol 63 No 8, Aug 90 (manuscript received 30 May 89) pp 1694-1697

[Article by A. L. Okley, T. A. Chubinidze, and G. G. Arevadze, Metallurgy Institute, GSSR Academy of Sciences]

[Abstract] Complex alloys that have a total silicon and manganese content of at least 75 atm% and that include various combinations of such elements as calcium, barium, aluminum, titanium, and boron are highly effective deoxidizing agents, microalloying agents, and modifiers of steel and cast iron. The study reported here generalized the results of previous studies of the thermodynamics of reduction in the complex systems Mn-Si-E-C-O (E being an element such as calcium, barium, titanium, aluminum, etc.) with an allowance for a number of inaccuracies that were previously allowed when representing the reduction process, specifically the reactions of the simultaneous reduction of oxides with the actual ratios of the oxide components of blends. The following six systems were examined: Mn-Fe-C-O, Si-Mn-Fe-C-O, Si-Mn-Ti-Fe-C-O, Si-Mn-Ca-Fe-C-O, Si-Mn-Ba-Fe-C-S-O, and Si-Mn-Ca-Ba-Al-Fe-C-O. Among other things, it was found that when the silicon content in an alloy of the system Si-Mn-Ca-Ba-Al-Fe-C-O (which qualifies as a complex silicon- and manganese-based alloy with respect to composition) is greater than 64 atm%, aluminum oxide is the oxide that is hardest to reduce from a thermodynamic standpoint. Tables 4; references 5 (Russian).

UDC 621.357.7

Features of Process of Palladium Coating in Limeda PD-4 Electrolyte

917M0005H Moscow ZHURNAL PRIKLADNOY KHIMII in Russian Vol 63 No 8, Aug 90 (manuscript received 10 Apr 89) pp 1721-1726

[Article by S. A. Didzhyulis and P. A. Yuzikis] [Abstract] Ammine chloride electrolytes with organic

additives, which are distinguished by their comparative simplicity to manufacture, are usually used to produce palladium coatings in the USSR. The problem with the procedure is that adding one additive to improve one of the electrolyte's properties sometimes worsens another as, for example, is the case of sheen-producing additives. the addition of which to the electrolyte often causes the resultant coating to crack after a thickness of 2-3 µm. To circumvent this problem the Chemistry and Chemical Technology Institute of the LiSSR Academy of Sciences developed the Limeda PD-4 ammine chloride palladium-coating electrolyte. This new electrolyte makes it possible to produce crack-free coatings up to 10 µm thick. Because satisfactory technological results have been achieved when the Limeda PD-4 is used, the authors of the study reported here decided to study selected features of the cathode process and the hydrogen absorption of coatings produced by using Limeda PD-4 electrolyte. The studies were conducted by extracting polarization curves in a potentiodynamic mode at a seanning speed of 2 mV/s and by the method of potentiostatic switching (with a PI-50-1 potentiostat, PDP-004 potentiometer, and \$8-13 oscillograph with memory). A palladium-coated (coating thickness, 3 µm) platinum disk pressed into Teflon (area, 1 cm²) served as the working electrode, a palladium wafer with an area of 8 cm² served as the anode, and the standard electrode was made of silver chloride. Several important differences were observed when the processes of electroplating palladium in an ammine chloride electrolyte without additives and with the Limeda PD-4 additive were compared. In the Limeda PD-4 electrolyte palladium is precipitated at much more negative electrode potential (E) values than in the electrolyte without additives. Further, when palladium is precipitated from the Limeda PD-4 electrolyte onto a palladium-coated electrode, a much greater quantity of hydrogen is formed at the beginning of the electrolysis than is formed when an electrolyty without additives is used (this quantity of hydrogen was not found to depend on E). Immediately after the precipitation, the integral hydrogen content in palladium coatings produced in the Limec'a PD-4 electrolyte was about fivefold that in coatings produced without additives. After 1 day of exposure under natural conditions, however, the hydrogen content in the coatings diminishes and for practical purposes equalizes Figures 5; references 10: 5 Russian, 5 Western

UDC 542.93:662.73:666.924.1

Decomposition of Mechanically Activated Nepheline Syenites in Hydrochloric Acid Solutions

917M0005I Moscow ZHURNAL PRIKLADNOS KHIMII in Russian Vol 63 No 8 Aug 90 (manuscript received 17 Oct 89) pp. 1751-1756

[Article by A. S. Berger, V. V. Boldyrev, B. Mirzoyev, and Kh. Sifiyev, Solid-State Chemistry and Mineral Ore Refining Institute Siberian Department, USSR

Academy of Sciences, and Chemistry Institute TaSSR Academy of Sciences]

[Abstract] The further development of alumina production in the USSR will largely involve using nephelinecontaining ore owing to its wide availability, the profitability of its integrated processing, and the possibility of locating enterprises near its source. The study reported here examined the hydrochloric acid breakdown of the nepheline syenites of Central Asia by subjecting them to preliminary mechanical activation. First, x-ray analysis, reference data, and the results of other published studies were used to compare the composition of Central Asia's nepheline syenites with those found elsewhere. The nepheline syenites in question were found to consist of about 26 mass% nepheline and 60 mass% high-silica minerals (albite, microlite, orthoclase). The nepheline svenite was then subjected to mechanical activation in a model AGO-1 laboratory planetary mill in an air medium with a mass ratio of specimen and balls of 1:20 and with a centrifugal acceleration of the balls of 40 and 60 g. The hydrochloric acid decomposition was conducted at a temperature of 363-368 K for 30 minutes. It was found that varying the concentration of the hydrochloric acid solution is important for separation of the solid and liquid phases after the experiment. Rapid sedimentation and good filterability of the sediment are achieved when 5-6 N HCl solutions are used, even with the maximum degree of decomposition of the activated specimens where the sediments consist of about 90 mass% hydrosilica gel. The maximum degree of aluminum extraction for x-ray-amorphous activated product (i.e., about 90 to 92%) was achieved during mechanical activation at a centrifugal acceleration of 60 g with a 5-6 N HCl solution. Such a regimen was found to result in a high extraction of aluminum (91%), potassium (92%), and sodium (100%) with simultaneous release of silicon into the sediment in the form of hydrosilica gel. Figures 3, tables 3; references 8 (Russian)

UDC 774.8

Investigation of Kinetics of Color Development on Model Silver Bromide Crystals

917M0005J Moscow ZHURNAL PRIKLADNOY KHIMII in Russian Vol 63 No 8, Aug 90 (manuscript received 19 Dec 89) pp 1784-1789

[Article by I. I. Mikhalenko, A. A. Kruglyak, L. V. Krasnyy-Admoni, and V. D. Yagodovskiy, Friendship of the People University imeni P. Lumumba and Light-Sensitive Materials Plant]

[Abstract] Studying the dye formation mechanism when photographic material is developed is very important in better defining the role of different factors affecting the activity of the color development reaction and the qualitative characteristics of the resultant color image. The study reported here analyzed the kinetics of the color development reaction and the effect of oxidizing agents on the process. An AgBr suspension (25 mg) was used to

study the formation of indoaniline dye. The AgBr was placed in the dish of a photocolorimeter, and developing solution was added to fill it. The concentration of components in the solution (mol/l) was as follows: Nethyl-N-β-hydroxyethylparaphenylenediamine, 1·10⁻⁴; α-naphthol, 1.2·10⁻⁴; K₂CO₃, 3·10⁻³ (to create a pH of 10.0); and ethanol, 8·10⁻² (to dissolve the α-naphthol). The concentration of indoaniline dye was determined on the basis of optical density at a wavelength of 618 nm after equal time intervals as the solution was periodically stirred. The rate of a homogeneous reaction in the absence of AgBr was determined in parallel tests. Tests

were conducted with white light-exposed and nonexposed specimens of AgBr. The values of the kinetic constants were determined, and the amount of Ag in the primary development centers of polycrystalline AgBr were estimated based on an analysis of the autocatalytic mechanism of this process. The authors demonstrated that introducing oxidizing agents into the developing solution increases the rate of indoaniline dye formation. In the case of the optimal additive, i.e., KSK-3, the rate of the process at the photolytic centers increased by a factor of 6-7. Figures 2, tables 2; references 4: 3 Russian, 1 Western.

UDC 541.135

Anodic Behavior of Gold in Alkaline Cyanide Solutions Containing Sulfide Ions

907M0296G Novosibirsk IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKIKH NAUK in Russian No 3, May 90 (manuscript received 14 Aug 89) pp 51-55

[Article by R. Yu. Bek and G. V. Kosolapor, Institute of Solid-State Chemistry and Processing of Mineral Raw Materials, Siberian Department, USSR Academy of Sciences, Novosibirsk]

[Abstract] Studies were performed by using a previously described device for electrode surface renovation. The working electrode was the tip of a gold wire 0.5 mm in diameter pressed into a Teflon shell. The supplementary electrode was a platinum wire and the comparison electrode a saturated silver chloride electrode. The results indicated that sulfide ions can have a double influence on the process of dissolution of gold in a cyanide electrolyte. On the one hand, when the content of the sulfide compound on the surface of the electrode is close to a monolayer, it accelerates the process. When thicker sulfur-containing films are formed, the process of dissolution of the gold is passivated. Figures 2; References 13: 9 Russian, 4 Western.

UDC 691.33:666.296

Ceramic Coatings From Charge Containing Nonferrous Metallurgical Slag

907M02984 Moscow STEKLO I KERAMIKA in Russian No 7, Jul 90 pp 2-4

[Article by A. A. Pashchenko, V. A. Sviderskiy, M. Yu. Chop, and A. S. Volkov, Kiev Polytechnical Institute]

[Abstract] A study is made of the color and gloss of ceramic tiles made with nonferrous metallurgical slag and clay. Surface gloss was determined by two parallel and independent tests. Regression equations were derived that were adequate to the experimental data with a level of a significance of 0.05 and 60 degrees of freedom. Areas of maximum vitrification are determined for the five slags tested. Coatings containing high-iron slags are presently being manufactured. Figures 3; References 3: Russian.

UDC 621.039.73:666.1

Vitrification of Sulfate Chloride-Containing Radioactive Wastes in Electric Furnace

907M0298B Moscow STEKLO I KERAMIKA in Russian No 7, Jul 90 pp 5-6

[Article by I. A. Sobolev, F. A. Lifanov, S. V. Stefanovskiy, A. P. Kobelev, V. N. Zakharenko. N. D. Musatov, and N. V. Krylova]

[Abstract] The method of vitrification can be used to produce more thermally, chemically, and radiationstable products than can cementation or bitumen treatment of medium-level radioactive wastes. Electric bath furnaces with continuous operation have been developed and are now being tested for vitrification of radioactive wastes at the Radon Scientific-Production Association in Moscow. However, contents of over 3% sulfates and chlorides by mass cause problems of phase separation. This article presents the results of experimental processing of radioactive wastes containing up to 16% sulfate ions and 10% chloride ions by furnace processing of a paste charge with a moisture content of 20-25% at 1150°C with datolite concentrate, lyuberetsk quartz sand, and loam. Vitrification of sulfate- and chloridecontaining radioactive wastes in the presence of a carbon reducing agent is ineffective and requires the development of special sulfate-containing glasses to immobilize the wastes. Another solution is also possible: separation of liquid wastes by preliminary precipitation of sulfates with bivalent metal salts into two streams—a sulfate-free solution and the sulfate sediment. The sulfate-free solution can be treated by vitrification, while the sediment, which contains practically no sodium ions, is treated by bituminization, cementing, or vitrification. However, this significantly complicates the process, meaning that single-stage vitrification with special glasses is more promising. References 4: Russian.

UDC 666.1.036.5

Temperature Mode of Fabrication of Optical Fiber Blanks

907M0298C Moscow STEKLO I KERAMIKA in Russian No 7, Jul 90 pp 13-15

[Article by V. V. Vysochin and S. S. Titar, Odessa Polytechnical Institute]

[Abstract] Modified chemical vapor-phase deposition (MCVD) is widely used in the production of optical fiber blanks. This article presents a study of the temperature conditions of this process under nonstationary conditions. Studies were performed on a machine with a moving system of radial burners to heat a horizontal seeding tube 18 mm in diameter, 1.5 mm in wall thickness, and 600 mm in length. Pyrometers were rotated around the vertical axis to scan the instantaneous temperature field in the process zone by recording changes in local temperature over time. An estimate of the transverse deformation of the temperature field in the wall indicates that a significant portion of the tube can be classified as a thermally thin body, with the greatest temperature gradient in a narrow area in the vicinity of the zone of external heat input, although even here the temperatures at the boundaries of the wall differ by not more than 10%. The temperature field is generally stable over time after the initial period of rapid temperature increase. In the deposition zone the wall temperature is stable near the burners. The most significant factor for determining the boundary conditions of the

process is the rate of movement of the flame. The data obtained indicate that the unsteady thermal interaction of the supporting tube and the surrounding environment must be considered in the process of improving MCVD conditions. Practical measures for the stabilization of conditions may include additional heating of the end portion of the tube, additional heating of the gas mixture before entry into the operating portion of the tube, and disconnection of the burner during return travel. Figures 3; References 2: Russian.

UDC 621.74:666.85/89

Heat-Resistant Cast Stone From Slag

907M0298D Moscow STEKLO I KERAMIKA in Russian No 7, Jul 90 pp 15-16

[Article by I. I. Bykov, Zh. D. Bogatyreva, V. A. Bogno, and L. F. Lekarenko, Institute of Casting Problems, UkSSR Academy of Sciences and Donetsk Cast Stone Plant]

[Abstract] The present Soviet production of thermally stable cast stone covers only a small fraction of the demand. Therefore, investigation and development of a process for the production of thermally stable cast stone were undertaken at the Donetsk Cast Stone Plant. The basic component of the charge used is granulated silicomaganese slag from the Nikopolsk Ferroalloy Plant. Eight pilot-scale runs have been made that have produced about 10 tons of product, with the yield of useable product up to 90%. The process of crystallization of the cast material was studied at 700-950°C with isothermal holding times of 5, 10, and 20 minutes. Crystallization is supported by the addition of only 1-2% catalytic additive such as chrome ore. The cast stone tiles produced have a service life up to five times as great as basalt cast stone materials. Products of this material can be produced on existing production lines without additional capital investment. The economic effect of the production and use of 1 ton of heat-resistant cast stone products is about 1,000 rubles per year. Figure 1.

UDC 666.3/7:536.2.21

Amphibole Heat-Insulating Ceramic

907M0298E Moscow STEKLO I KERAMIKA in Russian No 7, Jul 90 pp 19-21

[Article by Yu. I. Goncharov, N. A. Kholodova, and N. Ye. Sergeyev, Belgorod Construction Materials Technological Institute imeni I. S. Grishmanov]

[Abstract] A model insulating material with good structural characteristics and heat-physical properties has been developed based on the system NaF-MgO-SiO₂, in which fluorrichterite Na₂Mg₆(Si₈O₂₂)F₂ crystallizes—a mineral of the amphibole group. Tablets of amphibole ceramic were produced by semidry pressing at 30 MPa. Na richterite was synthesized in the process of roasting

the products at 500-1,150°C. The ceramic is recommended for the lining and thermal insulation of equipment operating at up to 100°C and for use in high-temperature filters. Figures 3.

UDC 536.70

Thermodynamic Modeling of Precipitation of Silicon in Closed System Under High Pressure

907M0299A Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 25 Jul 88) pp 1349-1352

[Article by M. Yu. Andreyev, V. I. Belousov, V. I. Korneychuk, and N. S. Papkov]

[Abstract] A thermodynamic analysis is presented of the system Si-Cl-H under pressures of 1.01-202·10⁵ Pa, and the possibility is studied of precipitating silicon in a closed system at this vapor-gas pressure level. The thermodynamics of the precipitation of silicon were computed for Si_{sol}, H₂, HCl, SiCl₄, SiCl₃, SiCl₂, SiHCl₃, SiH₂Cl₂, SiH₃Cl, and SiH₄. It is shown that there is an range of system states (Cl/H>0.5, T>1,400 K, p about 101·10⁵Pa) where it is possible to precipitate epitaxial layers of silicon about 1 µm thick by changing the temperature of the closed system. This process requires a gas interval of about 1 cm wide in which the required quantity of silicon is accumulated. The absence of a gas flow allows the method to be used for epitaxy with solid placement of substrates. Figures 4; References 5: 1 Russian, 4 Western.

UDC 548.52:539.216.1:546.28

Growth Mechanism of Threadlike Silicon Crystal in Initial Stage

907M0299B Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 14 Jul 88) pp 1353-1357

[Article by A. A. Shchetinin, B. M. Darinskiy, O. D. Kozenkov, and V. A. Nebolsin, Voronezh Polytechnical Institute]

[Abstract] An experimental study is presented of the initial stage of growth of a threadlike silicon crystal in a flow-through chloride system using particles of copper and gold as initiator impurities. The crystals were grown in a mixture of silicon tetrachloride and hydrogen at molar ratios of 0.005 to 0.025 and temperatures of 1,300 to 1,380 K. Substrates carrying crystals about 1 mm or less in length were studied by scanning electron and optical microscopes. It was found that the formation of a cosine-shaped pedestal results from compression of the droplet of initiator metal of the changing crystallization front bereath the drop and is determined by the temperature at which the threadlike crystals are grown and the

temperature of the impurity. Activation energies of the process of crystallization beneath a droplet were determined for the system Si-Au to be E_a = 78.5 kJ/mol and for the system Si-Cu, Ea = 212.9 kJ/mol. Figures 4; References 8: 7 Russian, 1 Western.

UDC 546.28

Electrophysical Properties of Films of Amorphous Hydrogenated Silicon Obtained at High Temperatures

907M0299C Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 18 Jul 88) pp 1358-1361

[Article by A. G. Kazanskiy, O. I. Konkov, and Ye. I. Terukov, Physical Technical Institute imeni A. F. Ioffe, USSR Academy of Sciences]

[Abstract] A study is made of the influence of substrate temperature on the activation energy of conductivity, edge of absorption, dark, and photoconductivity in amorphous hydrogenated silicon films. It is found that the electrophysical parameters of a-Si:H films precipitated at over 250° C depend on the nature of the bonds between the hydrogen and the silicon atoms. The properties of films produced at over 420° C are determined by the uniformly distributed monohydride bonds in contrast to films precipitated at less than 420° C, the parameters of which also result from the Si-H, Si = H₂, and (Si = H₂)_n bonds in clusters. Figures 3; References 18: 2 Russian, 16 Western.

UDC 539.216.2:621.315.592.2

X-Ray Diffraction and Electron-Microscope Study of InGaAs/GaAs (001) Heterostructures Grown by Organometallic Compound Hydride Epitaxy Method

907M0299D Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 21 Jul 88) pp 1369-1373

[Article by T. S. Kuntsevich, T. S. Babushkina, R. V. Kudryavtseva, and A. E. Ovsetsina, Gorkiy State University imeni N. I. Lobachevskiy and Scientific Research Institute. In Physics and Technology]

[Abstract] Methods of X-ray two-crystal diffractometry and topography and optical and electron microscopy are used to study the morphology of the surface, crystalline perfection, dislocation structure, and deformation variation of layers of $In_xGa_{1-x}As$ (where x is not over 0.14), grown on substrates of GaAs (001) by the organometallic compound hydride epitaxy method as a function of the composition of solid solution and growth conditions. When the heterostructures are grown in trimethylgal-lium-triethylindium-arsine-hydrogen at 600 and 670 °C,

two kinetic growth areas are found that correspond to different levels of perfection of the layers. The variation of elastic and elastic deformation of the layers with In content in the solid solution is determined for these areas, and a relationship is established between deformations and growth figures. The limits of coherent growth in layers of $In_xGa_{1-x}As$ are found for values of x up to 0.09 on a substrate of CaAs (001). Figures 4; References 10: 6 Russian, 4 Western.

UDC 538.113:549.31:678.072

Thermoradiation Effects in Zinc Selenide

907M0299E Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 14 Jun 88) pp 1395-1397

[Article by S. S. Kasymova, Zh. K. Komilov, and D. M. Yudin, Tashkent State Medical Institute]

[Abstract] A study is made of changes in the EPRspectroscopic properties of zinc selenide as a result of various types of external effects, including preliminary treatment by heat or heat and radiation. Specimens were heated to 500°C and held for 2, 3, and 4 hours, or sufficient power was applied to the furnace to heat it to 450°C, and then the additional heating to 500°C was acheived by exposure to a y field with a power of 1,600 R/s, thus combining heating and y irradiation of the specimens. Mechanical destruction of the specimens and subsequent heat and radiation treatment of the powders produced in air and in sealed ampules were used to estimate the influence of air on the optical properties of the material. It was found that the stability of the optical characteristics of ZnSe depends essentially on the surface condition. Figures 2; References 3: Russian.

UDC 621.315.592

Oriented Growth of TlInSe₂ Crystals

907M0299F Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 29 Jun 88) pp 1401-1404

[Article by I. V. Alekseyev, Institute of Physics, AzSSR Academy of Sciences]

[Abstract] Information is presented on the property of self-orientation found in the compound TlInSe and the use of this property for the oriented growth of single crystals. A tendency toward self-seeding is observed upon repeated recrystallization with temperature gradients. This method allows the growth of single crystals with the c axis oriented along an ingot, thus helping to increase the economy of the manufacture of crystals, improve their homogeneity, and preserve the high photosensitivity of the material. The self-seeding process can be described by the use of concepts of anisotropic growth

speeds and geometric selection in crystallization of a melt. Figures 3; References 7: 4 Russian, 3 Western.

UDC 546.26-162

Influence of Addition of Boron Oxide and Nitride on Morphology and Physical Properties of Graphite Foam

907M0299G Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 28 Apr 88) pp 1436-1440

[Article by A. A. Chuyko, V. N. Bykov, V. V. Dyakin, A. V. Gette, V. S. Yefanov, A. V. Melezhik, V. M. Ogenko, M. A. Tanatar, and V. V. Teslenko, Institute of Surface Chemistry, UkSSR Academy of Sciences]

[Abstract] A study is made of the influence of the addition of boron oxide and nitride on the morphology and physical characteristics of foamed graphite. Specimens were made of GT-1 and GSM-1 graphite with a flake size of 0.25-0.5 mm following oxidative heating in a sulfuric acid solution and subsequent heat treatment at 500°C. Some specimens were heat treated in the presence of boron oxide. Specimens of foamed graphite containing boron oxide were heated in an atmosphere of ammonia with the temperature gradually rising from 400 to 1,200°C over 6-7 hours to convert the oxide to nitride. The introduction of boron oxide and nitride to the foamed graphite merely changed the morphology of the specimen without influencing the graphite compounds present. A preferential accumulation of additives at the boundaries of crystals was also not observed. The foaming process facilitates changes in electron structure of the material apparently by preserving the residual sulfates after heat treatment. Figures 3; References 4: 3 Russian, 1 Western.

UDC 546.26-162

Synthesis and Properties of Fluorinated Carbon With Bromium Fluorides

907M0299H Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 6 Jul 88) pp 1441-1445

[Article by A. M. Danilenko, A. S. Nazarov, I. I. Yakovlev, O. G. Potapova, and V. P. Fadeyeva, Institute of Inorganic Chemistry, Siberian Department, USSR Academy of Sciences, and Novosibirsk Institue of Organic Chemistry, Siberian Department, USSR Academy of Sciences]

[Abstract] A study was performed of the products of the interaction of certain carbon-containing materials with bromium fluorides. Natural type GSM-1 graphite was used. Reactions were performed at 298 K. The particle size of the natural graphite was found to influence the

composition of the fluorinated graphite compounds formed. There was a correlation between d_{002} of the carbon-containing material and the degree of fluorination and quantity of bromium fluoride contained in the product. The method of producing carbon-containing materials can influence the composition of the fluorinated carbon materials formed. Figures 4; References 6: 5 Russian, 1 Western.

UDC 546.18

Surface-Stimulated Conversion of White Phosphorus Vapor to Nonvolatile Red Phosphorus

907M02991 Moscow IZVESTIYA AKADEMH NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 20 Jul 88) pp 1455-1458

[Article by I. V. Melikhov, O. V. Necheporenko, S. S. Berdonosov, and I. A. Kopylova, Moscow State University imeni M. V. Lomonosov]

[Abstract] When white phosphorus vapor is heated to 200-400°C. nonvolatile red phosphorus is formed by both surface and volumetric processes. A method of separating surface and volumetric phase segregations was used to isolate surface phase segregations in pure form. Analysis of the distribution function of amorphous red phosphorus particles precipitated from glass heated to 200-400°C by particle dimensions indicates that the growth centers of red phosphorus particles are seeds containing one or two unpaired electrons. The formation of red phosphorus particles from the vapor occurs by a mechanism of surface-stimulated polymerization. Figures 3: References 5 (Russian).

UDC 621.315.592+546.56:546.654:546.87

Production Conditions, Phase Composition and Physicochemical Properties of Specimens in System La₂CuO₄-Bi₂CuO₄

907M0299J Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 28 Jun 88) pp 1495-1498

[Article by L. A. Tikhonov, I. F. Kononyuk, Yu. G. Zonov, and L. V. Makhnach, Belorussian State University imeni V. I. Lenin; Scientific Research Institute of Physical and Chemical Problems]

[Abstract] A study was made of the system La_2CuO_4 -Bi $_2CuO_4$, in which a number of current-conducting materials can be expected with a broad range of electrical characteristics and superconducting properties. It was found that there are solid solutions $La_{2,x}Bi_xCuO_4$ with x between 0 and 0.1 and with x between 1.8 and 2.0. At x = 1.3-1.4, solid solutions such as $(Bi_2O_3)_{1,x}(La_2O_3)_x$ and copper oxide predominate. As x increases to 0.1 the conductivity of the specimens increases by almost an

order of magnitude, but no transition to superconductivity at over 4.2 K is observed. The room temperature resistivity of $\mathrm{Bi}_2\mathrm{CuO}_4$ varies between 10^2 and 10^6 Ohm m depending on the nature of the initial reagents and the production conditions. The coefficient of thermal expansion of $\mathrm{Bi}_2\mathrm{CuO}_4$ at 300-800 K is (5-6) 10^{-6} K⁻¹, increasing to 10-11- 10^{-6} K⁻¹ at 850-1,000 K. Figures 2; References 10: 6 Russian, 4 Western.

UDC 539.32

Mechanical Hardening of Lithium Iodate Single Crystals in Laser Radiation

907M0299K Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 6 Aug 88) pp 1526-1528

[Article by L. V. Atroshchenko, and N. V. Khodeyeva, Monokristallreaktiv Scientific-Production Association]

[Abstract] Data were obtained on the local structural and phase transitions when the (0001) face of an LiIO₃ single crystal was exposed to a single-mode pulse of neodymium laser radiation at an energy slightly over the threshold value. The pulse length was 15 ns. Microhardness H was studied as a function of distance from the center of the strike. Maximum hardening of the crystal was observed in the immediate vicinity of the center of the breakdown zone. Possible reasons for mechanical hardening of the single crystal by laser radiation are discussed, including local structural and phase transformations of the material upon heating and rapid cooling. Figures 2; References 8 (Russian).

UDC 621.315.592

Heat Conductivity of In_{1-x}Ga_xAs Solid Solutions

907M0299L Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 6 Jun 88) pp 1548-1550

[Article by M. I. Aliyev, D. G. Arasly, R. N. Ragimov, and Kh. A. Khalilov, Institute of Physics, AzSSR Academy of Sciences]

[Abstract] A study is made of the heat conductivity of an In_{1-x}Ga_xAs solid solution where x is between 0 and 0.1 in the 80-300 K temperature interval. Heat conductivity was measured by an absolute stationary method. Single crystals had an electron concentration 3.9·10¹⁷ cm⁻³. It was found that as the content of the second component increased the heat conductivity decreased and the curves

of $\kappa(T)$ become flatter. The concentration of electrons remains practically constant, conductivity decreases by a factor of 1.2, and electron heat conductivity is less than 2% of the total heat conductivity. Therefore, the decrease in heat conductivity and the temperature variation of κ are related to phonon processes. Figures 2; References 7: 3 Russian, 4 Western.

UDC 621.315.592

Jump Conductivity in TlGaSe₂ Single Crystals

907M0299M Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 15 Mar 88) pp 1555-1556

[Article by S. N. Mustafayeva and M. M. Asadov]

[Abstract] A study is made of the mechanism of charge transfer in TlGaSe₂ single crystals at low temperatures. The monotonically decreasing conductivity of TlGaSe₂ at low temperatures is explained by a conductivity mechanism based on localized states with variable jump length. According to this mechanism, low temperature conductivity results from jumps of charge carriers among states in a narrow band of energies near the Fermi level. The temperature-independent conductivity observed in TlGaSe₂ is considered by the authors to be nonactivation jump conductivity. Figure 1; References 5: 2 Russian, 3 Western.

UDC 535.232

Main Indices of Refraction of α-KY(WO₄)₂-Er³⁺ Crystals

907M0299N Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 7, Jul 90 (manuscript received 3 May 88) pp 1564-1565

[Article by N. N. Mitkin, A. A. Pavlyuk, R. A. Puko, A. P. Shkadarevich, V. V. Kuznetsova, and V. N. Verenik, Institute of Physics, BSSR Academy of Sciences, and BSSR Polytechnical Institute]

[Abstract] Polarization transmission spectra are used to determine the main indices of refraction of α -KY(WO₄)₂-Er³⁺ crystals. The polarized transmission spectra were recorded on a dual-beam spectrophotometer with a photometric error of 0.5%. The transmission spectrum of the crystal is a superimposition of continuous and line spectra, the latter belonging to the activator ion—erbium. The following results were obtained: $n_g = 2.15$, $n_p = 2.04$, and $n_m = 2.07$. Figure 1; References 5 (Russian).

UDC 541.183.546.2

Heterophase Impurity Inclusions in Optical Fibers of Chalcogenide Glasses

907M0315K Moscow VYSOKOCHISTYYE VESHCHESTVA in Russian No 4, Jul-Aug 90 p 192-197

[Article by G. G. Devyatykh, Ye. M. Dianov, V. G. Plotnichenko, I. V. Skripachev, M. F. Churbanov, and V. A. Shipunov, Institute of Chemistry of Highly Pure Substances, USSR Academy of Sciences, Gorkiy, and Institute of General Physics, USSR Academy of Sciences, Moscow]

[Abstract] Results are presented from studies of heterophase impurity inclusions in As-S and As-Se chalcogenide glasses and optical fibers based on these glasses. Optical losses to absorption and scattering in the glasses on impurity inclusions are computed and compared with the total losses in optical fibers. The studies indicate that chalcogenide glasses contain heterophase inclusions of submicron dimensions, their concentration depending on the method of fabrication of the glasses and varying within limits of 10⁵-10⁹ cm⁻³. An estimate of the optical losses in optical fibers of these glasses resulting from impurity heterophase inclusions demonstrates that in the long-wave area they are significantly less than the experimentally measured total optical losses. Figures 3; References 13: 9 Russian, 4 Western.

UDC 546.19(22+23).6-484+539.412.1

Strength of Optical Fibers Based on Chalcogenide Glasses Obtained by Crucible Method

907M0315L Moscow VYSOKOCHISTYYE VESHCHESTVA in Russian No 4, Jul-Aug 90 (manuscript received 11 Mar 90) pp 208-214

[Article by Ye. M. Dianov, V. M. Krsteva, V. G. Plotnichenko, S. L. Semenov, I. V. Skripachev, and M. F. Churbanov, Institute of General Physics, USSR Academy of Sciences, Moscow, and Institute of Chemistry of Highly Pure Substances, USSR Academy of Sciences, Gorkiy]

[Abstract] Results are presented from a study of single-layer and two-layer chalcogenide optical fibers with no coating and with a polymer coating after fabrication by the crucible method. The drawing was performed in a current of inert gas to protect the surface of the fibers from oxidation. Coatings were made of F-42 fluoro-plastic applied during drawing. In many cases two additional polymer coatings of polyvinyl chloride and polyacrylate were applied, the thickness of the triple polymer coating about 100 µm. A dynamic two-point bending method was used to measure the tensile strength and parameters of static fatigue strength. The mean values of bending strength at room temperature in air and liquid nitrogen were 932 MPa and 1.3 GPa for the F-42-coated fibers. Figures 4; References 10: 1 Russian, 9 Western.

UDC 621.315.592:535.213

Influence of Electron Bombardment on Optical Losses of Optical Fibers of Chalcogenide Glasses

907M0315M Moscow VYSOKOCHISTYYE VESHCHESTVA in Russian No 4, Jul-Aug 90 (manuscript received 13 Mar 90) pp 215-216

[Article by L. A. Litinskaya, N. V. Radygina, I. V. Skripachev, and V. A. Shipunov, Moscow Physics Engineering Institute, and Institute of Chemistry of Highly Pure Substances, USSR Academy of Sciences, Gorkiy]

[Abstract] A study is made of optical fibers with cores of As₄₀S₆₀ glass and jackets of As₃₅S₆₅ glass. The fibers were drawn from highly pure chalcogenide glass melts by a double crucible method. The core diameter was 180 µm; jacket diameter, 550 µm. Protective polymer jackets of F-42 fluoroplastic 10 µm thick plus polyvinylcloride, 500 μm, and polyacrylate up to 50 μm thick were applied. The increase in optical losses in the fibers under the influence of bombardment by fast electrons produced by a linear accelerator with an energy of 4.5 MeV, pulse length of 3 µm sec, repetition frequency of 400 Hz was studied. The total density of the electron flux was 5 1011 cm-2 s-11 t. A comparison of the results with previous works indicates that for equivalent bombardment doses the induced ontical losses are more than two orders of magnitude less, possibly a result of the higher purity of the glasses used in the fibers. Figure 1; References 10: Russian.

UDC 662.961.9

Investigation of Process of Obtaining Hydrogen From Siloxen

917M0005K Moscow ZHURNAL PRIKLADNOY KHIMII in Russian Vol 63 No 8, Aug 90 (manuscript received 18 Jan 89) pp 1802-1806

[Article by R. V. Chernov, Materials Science Problems Institute, UkSSR Academy of Sciences]

[Abstract] The method of obtaining hydrogen from siloxen is a new and promising direction in gas power engineering. This article reports a study examining the conditions of isolating siloxen from technical-grade alloys and its reaction with solutions of weak alkalis and presents hypotheses regarding its structure and possible areas for its practical application. The authors reacted Si₆H₆O₃ with 0.1 N NaOH (10 ml) to evolve hydrogen (sample weight, about 4 g; temperature, 22°; pressure, 750 mm Hg). The speed of the reaction turned out to be rather high and to be a function of the duration of the "aging" of the siloxen before it was used in the experiment. The freshly prepared specimen (aged for half an hour) yielded up to 99.5 vol% hydrogen after 10 minutes. whereas it took 24 hours for the entire volume of gas to be evolved. As the "aging" time increased, the initial reaction speed fell although the decrease in reaction time was insignificant within the bounds of an entire day. After 3 days of aging, only 67.2 vol% hydrogen was evolved. The overall hydrogen yield for specimens stored for 24 hours changed little, although there was a slight decrease of about 0.5-0.6 l/g. Soda, potash, and even solid wastes from selected industries were found to be suitable for use as reagents. The authors concluded that the hydrogen obtained by the proposed method may be used in many traditional applications areas (for example, as a fuel in organosilicon synthesis and to produce metal hydrides). They further concluded that the new method is especially promising for instances requiring the production of large volumes of gas in a short amount of time, in which case the conventional methods are slower and more costly. Tables 2; references 13: 5 Russian, 8 Western.

UDC 666.3:542.915:62-416:546.65'562'431

Producing Y-Ba-Cu-O Coatings by Pyrolysis of Aqueous Nitrate Solution

917M0005L Moscow ZHURNAL PRIKLADNOY KHIMII in Russian Vol 63 No 8, Aug 90 (manuscript received 12 Jan 90) pp 1813-1816

[Article by L. F. Chepik and Ye. P. Troshina] [Abstract] The recent discovery of the high-temperature superconductivity of YBaCuO ceramic ignited a scientific search for the techniques of producing this material not only in the form of bulk specimens but also in the form of coatings. The authors of the study reported here used the x-ray phase analysis method to study the effect of thermal conditions during the spraying and deposition of aerosol particles on the change in phase composition in a thin layer of coating. Their goal was to optimize the

conditions of obtaining the material in a specified crystalline state. A YBaCuO (123) coating was synthesized during pyrolytic decomposition of the aerosol formed by using an air stream to spray an aqueous solution of Y, Ba, and Cu nitrates. The coating was formed as the aerosol particles came into contact with the heated (to 300-600°) surface of the specimen owing to precipitation of a dry residue that had become enmeshed with its surface. The concentration of the aqueous solution of Y. Ba, and Cu nitrates was taken in a stoichiometric 1:2:3 ratio, which for Ba₂Cu₃O_x amounted to 122.25 g/l. An Al₂O₃ wafer served as the base. After being sprayed with the solution, the coated specimen was held for at least 2 hours at 600°. This was followed by further heat treatment of the specimen at 50° intervals from 600 to 1,000° with exposure for 2 hours at each fixed temperature and subsequent slow cooling to room temperature. The coating specimens were each at least 20 µm thick. A DRON-2.0 diffractometer was used to study the processes occurring in the coating layer. It was discovered that when the initial solution is sprayed the nitrates may decompose in one of two ways (modes A and B) that may be observed visually on the basis of the color of the resultant coating. Each is briefly discussed. The authors conclude that directed synthesis of a YBaCuO coating primarily in the form of the rhombic modification of the compound 123 requires pyrolytic decomposition of solutions of the aerosol nitrates under stable temperature conditions making it possible to avoid the intermediate compound BaCO₃. This is because the decomposition of BaCO₃ and subsequent formation of the compound 123 requires significantly higher temperatures. Figures 2; references 4 (Western).

UDC 541.124+547.314

Interaction of Tertiary Phosphines With Phenylacetylene With Proton-Donor Reagents Present

907M0288A Leningrad ZHURNAL OBSHCHEY KHIMII in Russian Vol 60 No 5, May 90 (manuscript received 22 Sep 88) pp 977-983

[Article by S. K. Barsegyan, G. Ts. Gasparyan, M. Zh. Ovakimyan, and M. G. Indzhikyan, Institute of Organic Chemistry, ArSSR Academy of Sciences, Yerevan]

[Abstract] Continuing previous studies in which adducts of 1:1 triakylphosphines with alkoxyphenyl-, diphenyl-, and isopropenylacetylenes were produced, the authors used an adduct of tributylphosphine with diphenylacetylene to find a new path to the formation of phosphobetaines that consisted of deprotonation of the corresponding quarternary phosphonium salts with triethylamine. This article describes the production of \beta-phosphobetaine from tributylstyrylphosphonium bromide. Diphenylmethylphosphine was used to produce a diphenylmethylphosphonium betaine with similar structure in a reaction with phenylacetylene, but the reaction product obtained was phenylmethyl(2-phenylstyryl)phosphine, apparently the product of intramolecular nucleophilic substitution of the phosphobetaine formed as an intermediate. The interaction of tertiary phosphines with phenylacetylene in the presence of proton-donor reagents resulted in the detection of anionotropic alkyl migration in compounds of 4substituted phosphine and a new direction for the reaction with breaking of the C-P bond after alkyl migration. Noncatalytic methods were developed for the production of styrylphosphonium salts. References 11: 6 Russian, 5 Western.

UDC 546.185'131+547.567.2

Autocatalytic Reaction of Phosphorus Pentachloride With p-Quinones

907M0288C Leningrad ZHURNAL OBSHCHEY KHIMII in Russian Vol 60 No 5, May 90 (manuscript received 16 Nov 88) pp 990-995

[Article by A. A. Kutyrev, S. G. Fomin, and V. V. Moskva, Kazan Institute of Chemical Technology imeni S. M. Kirov]

[Abstract] PCl₅ reacts with 1,4-benzoquinone at room temperature after a long induction period. The reaction is accompanied by an exothermic effect and the formation of an abundant dichlorophosphoran precipitate. When dichlorophosphoran is treated with water or alcohol, it is converted to the phosphate, which is also obtained by reverse synthesis by reacting PCl₅ with chlorohydroquinone in the presence of water. Phosphorus pentachloride reacts similarly with 2-chloro-1,4-benzoquinone. The end product of this reaction is a dichlorophosphoran, which is converted to two different compounds depending on external conditions. Phosphorus pentachloride reacts with

those p-quinones that attack hydrogen chloride with the formation of the corresponding chlorohydroquinones. The basic products of the reaction are the dichlorophosphorans. The process is autocatalytic and requires initiation. The authors suggest that tetrasubstituted p-quinones as well as 1,4-naphthoquinone, one of the closest structural analogues to 1,4-benzoquinone, do not interact with phosphorus pentachloride because they cannot attach hydrogen chloride. References 7: 1 Russian, 6 Western.

UDC 547.26'118+541.11

Thermochemistry of Organophosphorus Compounds. IV. Enthalpies of [1+4]-Cycloattachment of Phosphites to Benzyl in Solution and in Gas Phase

907M0288D Leningrad ZHURNAL OBSHCHEY KHIMII in Russian Vol 60 No 5, May 90 (manuscript received 16 Jan 89) pp 997-1001

[Article by V. V. Ovchinnikov, Yu. G. Safina, and R. A. Cherkasov, Kazan State University imeni V. I. Ulyanov-Lenin]

[Abstract] The thermal effects of the cycloattachment to benzyl of acyclic, 6- and 5-member cyclic phosphites in para-xylene at 35-50°C were determined, and the heats of the interaction were computed for the gas phase. The enthalpies of solvation of the phosphites and benzyl are presented in a table. A study of the thermochemistry of the process of [1+4]-cycloattachment of phosphites to benzyl in solution and in the gas phase establishes that the reactivity of phospholan derivatives is significantly less than that of the 6-member analogues due to the higher internal ring energy. References 30: 26 Russian, 4 Western.

UDC 547.241

Phosphorus-Containing Ketenes as Reagents for Intramolecular Cyclization

907M0288G Leningrad ZHURNAL OBSHCHEY KHIMII in Russian Vol 60 No 5, May 90 (manuscript received 19 Dec 88) pp 1050-1053

[Article by O. I. Kolodyazhnyy, Ye. V. Grishkun, V. I. Yakovlev, and V. P. Kukhar, Institute of Bioorganic Chemistry, UkSSR Academy of Sciences, Kiev]

[Abstract] Phosphorus-containing ketenes are used as reagents for intramolecular cyclization accompanied by splitting of a phosphoryl group. Attachment to a phosphorus-containing ketene of a nucleophil containing a mobile hydrogen atom and carbonyl group yields an attachment product that, as a result of an intramolecular Horner-Witting reaction, is converted through an intermediate carbanion to a cyclic product. The reaction is usually conducted in two stages. References 16: 9 Russian, 7 Western.

UDC 543.5+632.95

Determination of Mixtures of Pesticides Used in Treatment of Grain Seeds by TLC and HPLC

907M0296F Novosibirsk IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKIKH NAUK in Russian No 3, May 90 (manuscript received 1 Aug 89) pp 20-24 [Article by Ye. Yu. Kozlova, Novosibirsk Institute of Organic Chemistry, Siberian Department, USSR Academy of Sciences]

[Abstract] Fungicide levels are determined in mixtures of various compositions by TLC and HPLC in wheat and barley seeds following pesticide treatment. The pesticides were extracted from the seed material in ethyl acetate. Results obtained by the two methods are compared. Figures 4; References 17: 13 Russian, 4 Western.

UDC 553.981.982.041(477.9)

Spreading of Hydrocarbon Deposits in Black Sea-Crimean Region

907M0295A Kiev NEFTYANAYA I GAZOVAYA PROMYSHLENNOST in Russian No 3, Jul-Sep 90 (manuscript received 01 Dec 88) pp 2-5

[Article by R. M. Novosiletskiy, A. G. Ilkiv, and A. Yu. Polutranko, UkSSR Scientific Research Institute of Geological Prospecting]

[Abstract] Analysis of geological, geochemical, and fluid dynamic data on the Black Sea-Crimean oil and gas region is used to generate an area and depth prediction of the extent of pays and deposits of hydrocarbons. The basic factor involved in the upward migration of fluids is the relief of anomalously high pressure primarily of methane and water squeezed out in the process of lithification and thermocatalytic decomposition of organic compounds, including high-molecular mass hydrocarbons. Release of fluids with anomalously high pressure in deposits in the late catagenesis stage leads to the formation of hydrocarbon deposits in overlying rock. As a result of this, the Southern Black Sea-Crimean area contains a thick mass of slightly transformed Mesozoic-Cenozic deposits that are intensely generating hydrocarbons, thus facilitating the formation of deposits upon release of fluids with anomalously high pressure in the peripheral areas of the Black Sea depression. Large deposits of hydrocarbons at moderate and great depths can be expected in the Black Sea region and the Southeastern portion of the Kerchenskiy Peninsula. Figures 2; References 4 (Russian).

UDC 551.242:550.812.1:553.98

Basement Relief and Prospects of Beyevo-Akhtyrskaya Area

907M0295B Kiev NEFTYANAYA I GAZOVAYA PROMYSHLENNOST in Russian No 3, Jul-Sep 90 (manuscript received 21 Feb 89) pp 5-9

[Article by V. I. Yakimovich, V. F. Shulga, and V. I. Kaledina, UkSSR Scientific Research Institute of Geological Prospecting, Institute of Geological Sciences, UkSSR Academy of Sciences]

[Abstract] A combined study method involving graphic approaches to refine the tectonics, study of the specifics of basement relief, construction of its hypsometry, and determination of objects promising for oil and gas deposits was applied to the 1,200 km² Beyevo-Akhtyrskaya area. The method of studying the basement relief used is founded on an analysis of a series of specially constructed maps: faults and their systems, satellite-tectonic, isolines of geophysical parameters (gravitation, magnetic fields, total conductivity), thicknesses of various sedimentary complexes and structural surfaces, and schematic geologic maps of the base of the platform cover and the Precambrian level. Gravimetric

data and the discovery of areas of reduced boundary velocities by the refracted wave correlation method have revealed a number of areas that appear promising in the search to find hydrocarbon traps in the Precambrian bed. They should be prospected after the Khukhrinskiy deposit is studied to produce additional data on the details of its basement trap conditions. References 9: Russian.

UDC 553.98:622.276

Evaluation of Gas Reserves Based on Production Dynamics

907M0295C Kiev NEFTYANAYA I GAZOVAYA PROMYSHLENNOST in Russian No 3, Jul-Sep 90 (manuscript received 18 May 88) pp 9-11

[Article by A. A. Lagutin, A. V. Lizanets, and V. Yu. Rogozhin, UkSSR Scientific Research Institute of Gas]

[Abstract] A study is made of the gas content of the Kremenovskiy deposit. Previous studies by the volumetric method and the method of decreasing pressure have not yielded consistent results. Therefore, the dynamics of gas production was analyzed for the entire period of exploitation of the deposit since 1977 by studying the variation in annual and total gas production and considering the drilling of new wells. It was assumed that the entire gas-bearing volume of the deposit is being drained by the existing wells. Equations are derived that indicate that the deposit initially contained 2-4% more gas than is indicated by the method of decreasing piessure and 37-40% more than was initially indicated by volume studies. Detailed analysis of gas production dynamics in late stages of exploitation of deposits thus allows a computation of the initial gas reserve even when information is not available on the geological structure of the reservoir or the pressure in the deposit. The method can be used independently or in combination with the method of decreasing pressure. Figures 2.

UDC [553.98.551.73](477.7)

New Preddobrudzhinskiy Depression Oil and Gas Area

907M0295D Kiev NEFTYANAYA I GAZOVAYA PROMYSHLENNOST in Russian No 3, Jul-Sep 90 (manuscript received 7 Jun 89) pp 11-13

[Article by B. I. Denega, R. V. Palinskiy, and V. N. Ablikov, UkSSR Scientific Research Institute of Geological Prospecting, Krymgeologiya Geophysical/Production Association]

[Abstract] The Paleozoic Preddobrudzhinskiy depression in the western Black Sea area represents more than one third of the potential hydrocarbon resources of the southern Ukraine. The Paleozoic deposits represent 70% of the resources predicted for the depression, and the productive and promising strata are at 2,600-3200 m

depth, an indication of the great promise of the area. This article discusses the specifics of the geological structure and oil content of the eastern Saratskiy deposit in the Devonian strata. A method is suggested for conducting prospecting operations in other parts of the area with similar structure. Figure 1.

UDC 622.24.082:622.243.92

Planning and Analysis of Turbine Drilling Conditions

907M0295E Kiev NEFTYANAYA I GAZOVAYA PROMYSHLENNOST in Russian No 3, Jul-Sep 90 (manuscript received 2 Dec 88) pp 21-23

[Article by L. I. Dopilko, Poltava Directorate of Drilling Operations]

[Abstract] During turbine drilling, the load on the bit, rotating speed, and drilling solution feed rate are interrelated. Planning of drilling operations must involve a realistic calculation of the effect these parameters have on each other. Under real conditions, the feed rate of the solution is changed in discrete steps, and the load on the bit is frequently determined by process factors, particularly during drilling of slanting bore holes. The basic hydraulic characteristics of turbine drills are used in this article to determine the bit rotation speed with independent changes in load or solution feed or simultaneous changes in both parameters. A simple equation is derived that interrelates the load on the bit, its rotating speed, and the drilling solution feed rate. The method developed here for determination of the torque expended in rotating the bit was in the field, and the mean-square error was found to be not over 15%, with a confidence level of 0.9.

UDC 622.24.051.64

Testing of 292.9 mm-Diameter Cutter Bit

907M0295F Kiev NEFTYANAYA I GAZOVAYA PROMYSHLENNOST in Russian No 3, Jul-Sep 90 (manuscript received 26 May 89) pp 24-26

[Article by V. N. Romashov, Northern Caucasus Scientific Research Institute of Gas]

[Abstract] Between 1966 and 1988, the Karachaganak-skiy Gas-Condensate deposit was selected for testing of imported 292.9-mm-diameter diamond-tipped cutter bits. This deposit was selected because of the favorable geological conditions and the availability of the required equipment and trained personnel. The bits were used to drill a 244.5 mm column to a depth of 20-35 m in the roof of the Filippov stratum, usually by the turbine method. In most cases the lower Triassic, upper Permian, and a portion of the lower Cretaceous deposits were penetrated. An analysis of the geological conditions of drilling, the results of operation of the diamond cutter bits, and a comparison with tricone bits indicates that

the area tested is promising and favorable for extensive application of these bits. Monitor washing and erosion-resistant bodies are suggested for use in domestic bits of similar design. The geological cross section of the deposit should be divided into sections of identical drillability based on the results of testing with tricone or diamond bits. It is suggested that studies be continued, with a comparison of domestic and imported diamond cutter bits.

UDC 622.276(477.8)

Increasing Output of Low-Permeability Carpathian Oil Deposits

907M0295G Kiev NEFTYANAYA I GAZOVAYA PROMYSHLENNOST in Russian No 3, Jul-Sep 90 (manuscript received 5 Mar 90) pp 28-31

[Article by M. I. Buchlovskaya, J. I. Muzychko, and Yu. M. Smuk, Ukrneft Production Association]

[Abstract] The production of the Menilitovaya area of the Dolinskiy deposit is compared for separate blocks differing in a number of parameters including extraction, density of wells, flow rate per well, stratal pressure, and quantity of water injected. The studies indicate that in order to increase production, the grid of wells must be made denser by utilizing wells drilled into deeper strata for this purpose. Drilling of 17 new wells is also recommended in order to permit extraction of an additional 1.710 million tons of oil. Figure 1.

UDC 665.61.033.55:542.943

Role of Thermal Oxidation in Process of Extracting Vanadium From Refinery Coke (Solid Oil Residue)

917M0003A Moscow NEFTEKHIMIYA in Russian Vol 30 No 4, Jul-Aug 90 (manuscript received 17 Jan 90) pp 449-452

[Article by G. Yu. Sitnikova, N. V. Rastova, S. L. Davydova, R. D. Archakova, S. N. Khadzhiyev, and Yu. B. Amerik, Petrochemical Synthesis Institute imeni A. V. Topchiyev, USSR Academy of Sciences, Moscow, and Grozneftekhim Scientific Production Association] [Abstract] In view of the scarcity of vanadium on the world market, it is critical that other sources of vanadium besides ore be found. The vanadium concentration in heavy oils is comparable to that in ores, and the vanadium concentration in residual products of oil (mazout and bitumen) may be doubled by atmospheric distillation and quadrupled by vacuum distillation. Simply burning mazout and collecting the vanadiumenriched ash is, however, fraught with a number of problems. Consequently, the enrichment heavy oil residues and commercial separation of vanadium are interconnected, and solving them requires developing methods of concentrating the vanadium in solid oil residues and separating them from the liquid portion of

heavy oil residue. In the study reported here, the process of separating vanadium from solid oil residue was implemented by way of the example of cokes produced in the processes of delayed coking, thermal contact cracking, flexicoking, and thermolysis of bitumen and mazout. It was discovered that preliminary heat treatment of cokes in the presence of oxygen makes it possible to extract a high (80 to 85%) percentage of vanadium from coke. This is evidently due to two factors, namely, the concentration of vanadium and its transition into oxides that are easily released from the coke matrix when it is processed with sodium hypochlorite (NaOCI) in a sulfuric acid medium. The efficiency of the process developed was found to depend on the time and temperature of the process and on the rate at which air is fed into the reactor. Tables 3; references 8: 6 Russian, 2 Western.

UDC 547.732:542.941.7:661.183.6

Catalytic Activity of High-Silica Organosilicate and Aluminosilicate Zeolites in Reaction of Thiophene Hydrogenolysis

917M0003B Moscow NEFTEKHIMIYA in Russian Vol 30 No 4, Jul-Aug 90 (manuscript received 27 Jul 89) pp 482-485

[Article by A. V. Vysotskiy, V. I. Kistinev, Yu. G. Khaybulin, G. V. Kuznetsova, K. F. Kosygina, and I. 1e. Gorbatkina; Bratsk Industrial Institute, Bratsk: Petroand Coal-Chemical Synthesis Institute, Irkutsk State University, Irkutsk; and All-Union Scientific Research Institute of Petroleum Refining, Moscow]

[Abstract] Previously, the authors of this article established that an aluminum oxide matrix based on commercial Al(OH)₃ with a low content of active group VI (Mo) and group VII (N², Co, Fe) metals may be promoted by 5-10% high-silica boron and iron silicate zeolites. In their work published here the authors study AlMoNi catalysts promoted by organosilicates and aluminosilicates in the hydrogenolysis of thiophene, compare their catalytic activities, and examine the nature of the active centerpromoters. They discover that adding 10 mass% highsilica organosilicate zeolites (boror, iron, and chromium silicates) to an AlNiMo system results in a significant intensification of the hydrodesulfurization of thiophene, hydrogenation of the resultant C_4 -alkenes to n-butane, and a reduction of contacts' capability of splitting (cracking) the C-C bonds. It is concluded that these are the best catalysts for hydrofining light oil distillates. It is also found that adding 10 mass% aluminosilicate pentasils to an AlNiMo catalyst has a much less active effect on the hydrogenolysis of C-S bonds, with the hydrogenating activity remaining at the same level but with the cracking of the C-C bonds occurring more intensively. These catalysts are recommended for use in hydrofining heavy oil residues. Figure 1, table 1; references 4 (Russian).

UDC 659.6.097.3:662.183.6

Effect of Hot Steam Treatment of Pentasil-Containing Catalyst on Its Activity and Selectivity in Transformations of Delayed Coking Gasoline

917M0003C Moscow NEFTEKHIMIYA in Russian V 1 30 No 4, Jul-Aug 90 (manuscript received 15 Nov 89) pp 501-506

[Article by V. G. Karakashev, A. Z. Dorogochinskiy, and N. V. Shirokova, Grozdno Oil Institute imeni Academician M. D. Millionshchikov]

[Abstract] The reported studied here examined the effect of hot steam treatment of type TsVM (SiO₂/Al₂O₃ = 26) pentasil-containing catalyst on its activity and selectivity in transformations of delayed coking gasoline. The dependence of catalytic activity on the duration of hot steam treatment reaches its maxium at temperatures between 550 to 600°C (with the dependence being more expressed at 550°C than at 600°C). At higher treatment temperatures, catalytic activity increases as the duration of hot steam treatment decreases. As the duration of hot steam treatment increases, the catalyst's selectivity increases in the formation of gasiform products and C_2 - C_4 olefins, whereas its selectivity in the formation of aromatic hydrocarbons and coke decreases, eventually reaching a stable value that is identical for all of the hot steam treatment temperatures examined. It is concluded that hot steam treatment conducted at a temperature of 550°C for 160 minutes results in a catalyst that is among the most active and selective in the formation of C₂-C₄ olefins (up to 46 mass%). This time and temperature also improve the make-up of the liquid reaction products by reducing their concentration of unsaturated hycrocarbons and increasing their content of aromatic hydrocarbons. Figures 2, table 1; references 9 (Russian).

UDC 547.569:547.722

Formation of Vinyl Sulfides Under Effect of Dichlorocarbene on Alkyl(arylmethyl)sulfides Under Interphase Catalysis Conditions

917M0003D Moscow NEFTEKHIMIYA in Russian Vol 30 No 4, Jul-Aug 90 (manuscript received 31 Oct 89) pp 551-555

[Article by A. M. Al-Shura, N. V. Fedorov, A. V. Anisimov, and Ye. A. Viktorova, Moscow State University]

[Abstract] The study reported here examined the reaction of saturated sulfides containing benzyl (I, II) and 1-naphthylmethyl (III, IV) residues with dichlorocarbene generated under conditions of interphase catalysis. (Sulfides with this structure were introduced into this reaction since doing so expanded the scope of their synthetic application, for instance, to synthesize halogen-substituted unsaturated sulfides, which are difficult to produce by isomerization of the respective allyl sulfides

or by Pummerer's sulfoxide reaction.) When triethylbenzyl ammonium bromide was used as a catalyst, the effect of dichlorocarbene (obtained from chloroform in the presence of an aqueous solution of sodium hydroxide) on group I and II sulfides resulted in previously unknown unsaturated halogen-substituted sulfides. In the case of alkyl(1-naphthylmethyl)sulfides, an ethyl ether of 1-methyl-2-naphthalic-carbonic acid was formed. Tables detailing the percent yield and paramagnetic resonance and mass spectra of the compounds formed and the ¹³C nuclear magnetic resonance of the resultant alkylvinyl sulfides are presented. Tables 2; references 7: 3 Russian, 4 Western.

UDC 543.852

Method of Direct Determination of Free Strong Acids in Commercial Oils

917M0003E Moscow NEFTEKHIMIYA in Russian Vol 30 No 4, Jul-Aug 90 (manuscript received 6 Oct 89) pp 567-569

[Article by N. M.-G. Shvekhgeymer, Zh. A. Krasnaya, T. S. Stytsenko, and V. I. Slovetskiy, Organic Chemistry Institute imeni N. D. Zelinskiy, USSR Academy of Sciences, Moscow]

[Abstract] According to its authors, this article is the first to propose a direct high-sensitivity method of determining free strong acids in oils. The new method is based on a spectrophotometric analysis of solutions of oils in the presence of TsP-2 organic indicator and subsequent determination of the amount of acid by using a calibration chart. Work to develop the method was conducted on M-40 (Karl Zeiss) and UVIDEC (IASCO) spectrometers. Measurements were taken in the interval from 420 to 670 nm. Methylene chloride that had been specially cleaned to removed traces of strong acids was used as the solvent. The method developed was found to have a free acid detection limit of 10⁻⁷ mol/g. Statistical processing of the results obtained by using the new method indicated that it possesses good precision and reproducibility. In fact, the method proposed was found to be even more accurate than the method currently stipulated in the respective All-Union State Standard [GOST]. (This latter fact is likely due not to any systematic error inherent in the GOST-specified method but rather to the fact that the etherification of ethyl alcohol and other secondary reactions called for in the GOST consumes some of the acids in the samples undergoing analysis, thus resulting in a reduced acid content.) Tables 2; references 3 (Russian).

UDC 547.436:621.892.099.6:66.094.38

1,2-Aminopropanethiols as Lubricating Oil Additives

917M0003F Moscow NEFTEKHIMIYA in Russian Vol 30 No 4, Jul-Aug 90 (manuscript received 15 May 89) pp 570-573

[Article by M. A. Allakhverdiyev, A. T. Guseynova, V. M. Farzaliyev, Yu. M. Sultanov, and M. A. Gadzhiyeva, Additive Chemistry Institute, AzSSR Academy of Sciences, Bakul

[Abstract] It has previously been established that 1,2aminothiols with the general formula R-CH(SH)CH₂NHC₆H₅ and their transition metal complexes possess a high antioxidant activity, thus making them suitable for use as oxidation inhibitors in petroleum products. In a continuation of this research direction, the authors of the study reported here synthesized 1,2-aminopropanethiols containing aniline fragments and examined the effect that various electron donor and electron acceptor substitutes and their position in the benzene ring have on antioxidant activity. The authors established that 1,2-aminopropanethiols are effective antioxidant additives to petrolatum. They also discovered that it is the NH- and SH-functional groups in a molecule of 1,2-aminopropanethiols used as a petrolatum additive that are the main carriers of its antioxidant and antimicrobial properties. It was further found that all of the 1,2-aminopropanethiols synthesized inhibit oxidation of petrolatum and that, from an effectiveness standpoint, they are at the level of the commercial inhibitor 2,6-di-tert-butyl-4-methylphenol (ionol). Tables 2; references 8 (Russian).

UDC 678-9:66.09

Accelerated Light Aging of Cellulose Acetate Butyrate Films

917M0005M Moscow ZHURNAL PRIKLADNOY KHIMII in Russian Vol 63 No 8, Aug 90 (manuscript received 12 Feb 90) pp 1780-1784

[Article by T. L. Temchenko, E. S. Shulgina, and A. F. Nikolayev]

[Abstract] Polymer films produced from solutions of cellulose esters and used as optical materials undergo marked photodestruction under the effect of the ultraviolet component of natural or artificial light; this in turn results in a worsening of the physicomechanical and spectral properties and, consequently, the service life of the said films. The study reported here examined selected laws governing the occurrence of photodestruction processes in cellulose acetate butyrate (CAB) films, which are the basis of organic optical filters. Films were produced from a 7% solution of CAB in a chloroformethyl alcohol (85:15) mixture by pouring onto reflective glass bounded by a wooden frame. This is done by isothermal evaporation of the solvent. Dibutyl phthalate, in a quantity of 10% of the mass of the CAB, was added to the film-forming composite as a plasticizer. The films produced were 150 µm thick. They were aged by using a DRB-8 lamp, which has a maximum radiating intensity (82.8%) at a wavelength of 253.7 nm (which corresponds to the maximum destruction of CAB). It

was found that regardless of whether the CAB was in film or powder form, it underwent a sharp drop in characteristic viscosity during the first 10-15 minutes of irradiation, followed by a stabilization or only slight increase in the drop thereafter. After I hour of irradiation, the luminous transmission of both the plain CAB films and those to which dibutyl phthalate had been added stabilized; however, the plain CAB films had a luminous transmission that was 2% higher than that of CAB films to which dibutyl phthalate had been added (with the latter having a luminous transmission of 90-92% in the visible [500-800 nm] region of the spectrum). In the ultraviolet (250-350 nm) region the plain and dibutyl phthalate-containing films acted differently. In the optical spectrum of the nonplasticized films there was an absorption band at 280 nm (the intensity of which decreased noticeably as the duration of the specimen's irradiation increased). When dibutyl phthalate was added to the CAB film, the characteristic bands of the polymer and dibutyl phthalate were superimposed on the specimens' spectra. As a result the boundary of the beginning of transmission shifted from 240 to 290 nm, and as the duration of the irradiation increased, the appearance and development of an absorption band at 320 nm was noted. The scattering of light energy on the dibutyl phthalate reduced the photodecomposition of the CAB somewhat. It did not, however, provide complete protection against the destructive activity of light. Figures 3, table 1; references 7: 4 Russian, 3 Western.

UDC 546.791.2+546.791.3

Reduction of Trivalent Uranium to Bivalent State in Fused Salts

907M0290A Leningrad RADIOKHIMIYA in Russian Vol 32 No 2, Mar-Apr 90 (manuscript received 4 Apr 89) pp 1-4

[Article by N. B. Mikheyev, Ye. R. Merts]

[Abstract] A study is made of the possibility of reducing uranium to the bivalent state by the method of reducing a trace element in a chloride melt with a bivalent lanthanide. This method is based on a study of the distribution of a trivalent trace element between the solid neodymium oxychloride phase and a melt containing NdCl₂, NdCl₃, and SrCl₂. It is demonstrated that trivalent uranium is reduced to the bivalent state when bivalent neodymium is present in the melt. The standard oxidation potential is determined as -2.54 V. Figures 2; References 3: 1 Russian, 2 Western.

UDC 541.49:543.426:546.799.3

Luminescence of Neptunium Ion in Fluorite Crystal

907M0290B Leningrad RADIOKHIMIYA in Russian Vol 32 No 2, Mar-Apr 90 (manuscript received 3 Sep 89) pp 4-7

[Article by A. S. Babayev and R. B. Dushin]

[Abstract] The purpose of this work was to suggest a model of the structure of an impurity center based on more detailed analysis of the luminescence spectrum of Np in CaF₂ in the visible area of the spectrum. The bands in the luminescence spectrum are related to transitions to electron sublevels of the basic Np⁴⁺ (f³) level at the cubic symmetry substitution center. The parameters of the crystalline field are determined. An estimate is presented of the crystalline field parameters based on a model of point charges. The estimated values of the parameters for the Np⁴⁺ ion are in qualitative agreement with the observed values. Figure 1; References 7: 6 Russian, 1 Western.

UDC 546.661:539.125.5+546.662.027.05

Selection of Optimal Conditions of Accumulation of ¹⁵³Gd During Bombardment of ¹⁵¹Eu by Thermal Neutrons

907M0290C Leningrad RADIOKHIMIYA in Russian Vol 32 No 2, Mar-Apr 90 (manuscript received 21 Jul 89) pp 29-33

[Article by A. A. Nosov, A. D. Gedeonov, and B. N. Shuvalov]

[Abstract] The purpose of this work was to select by a calculation method the optimal conditions for bombardment of europium 151 in a flux of thermal neutrons to

produce gadolinium 153 with the maximum specific activity and minimum content of long-lived impurity radionuclides, which can significantly distort the natural γ spectrum of gadolinium 153. The maximum specific activity of ¹⁵³Gd and minimum content of long-lived impurity radionuclides is achieved by bombardment in a flux of thermal neutrons with a density of 1 10¹⁴ cm⁻²·s⁻¹ for 70 days. Figure 1; References 14: 6 Russian, 8 Western.

UDC 543.52

Radiometric Determination of Californium-252 in Solutions

907M0290D Leningrad RADIOKHIMIYA in Russian Vol 32 No 2, Mar-Apr 90 (manuscript received 4 Sep 89) pp 37-41

[Article by Yu. P. Anokhin, V. D. Gavrilov, Yu. V. Yefremov, Yu. S. Popov, V. B. Mishenev, and G. A. Timofeyev]

[Abstract] Methods are presented for determining the content of californium-252, including the method of measurement of the yield of neutrons upon spontaneous fission, the method of quantitative determination by measuring the intensity and spectrum of α radiation, and the method of isotope dilution and α spectrometry. Quantitative determination by measurement of the intensity and spectra of its α radiation has an error level of 2-3% and is used in analytic monitoring of process emissions and the purification of californium. Isotope dilution and α spectrometry has an error of 3-4%. Results are presented from determining the californium-252 content in a nitrate solution used for the manufacture of neutron sources and in prepared neutron sources by several methods. Figures 2; References 3 (Russian).

UDC 621.311.25

Leaching of Radionuclides From Nuclear Fuel Particles and Reactor Graphite Extracted From Specimens Within 30 Kilometers of Chernobyl

907M0290E Leningrad RADIOKHIMIYA in Russian Vol 32 No 2, Mar-Apr 90 (manuscript received 14 May 89) pp 55-59

[Article by V. A. Avdeyev, A. S. Krivokhatskiy, V. G. Savonenkov, Ye. A. Smirnova]

[Abstract] Finely dispersed (0.2-1.0 mm) particles of fuel and reactor graphite were separated from samples of sandy soil collected in the western fallout zone from the Chernobyl reactor in October of 1986. Samples were collected 0.5 to 2.0 km from the site of the accident. Individual fuel and graphite particles were selected by hand under a microscope. Leaching was performed by using an aqueous soil extract from the area of the power plant. It was found that the leaching of radionuclides from dense, hard fuel particles was lower than from

other types of fuel particles. A count of fuel particles in specimens after leaching indicated that their number was unchanged and their external appearance remained the same. References 2: Russian.

UDC 621.311.25

Leaching of Radionuclides by Solutions of Various Compositions From Soil Specimens Collected Near Chernobyl Nuclear Power Plant in 1986

907M0290F Leningrad RADIOKHIMIYA in Russian Vol 32 No 2, Mar-Apr 90 pp 59-64

[Article by V. A. Avdeyev, Ye. I. Biryukov, A. S. Krivokhatskiy, V. N. Selifonov, and Ye. A. Smirnova]

[Abstract] A study was made of the leaching of radionuclides from soil samples gathered in May of 1986 in the northwest and west radioactive fallout zones at 14.6 and 1.2 km from unit 4 of the Chernobyl Nuclear Power

Plant. Specimens were gathered by removing the surface layer of the soil about 1 cm thick from an area of about 0.3 m³. The soil specimens were leached by using aqueous extracts of fallen leaves, pine branches and needles, turf, and chernozem soil from the area around the power plant. Experiments were performed with a ratio of specimen weight to solution volume of 1 g/25 ml. Leaching was performed at room temperature and at 85°C, and the specimens were then centrifuged and analyzed for content of y-radiating radionuclides. In both specimens some of the radionuclides were lost during leaching, or their concentration was significantly decreased. The degree of transfer of radionuclides to the solution increased with increasing time and acid concentration of the solutions. Both cesium-137 and cerium-144 went into solution more easily from the specimen taken 14.6 km to the northwest of the power plant than from the specimen taken 1.2 km to the west, thus indicating the individuality of the specimens.

UDC 542.422.2:661.728

EPR Investigation of Radiation Chemical Destruction of Cellulose by Accelerated Electrons

917M0005N Moscow ZHURNAL PRIKLADNOY KHIMII in Russian Vol 63 No 8, Aug 90 (manuscript received 17 Oct 89) pp 1867-1869

[Article by F. M. Gumerov, V. A. Silayev, D. S. Nusinovich, Ye. L. Matukhin, and V. F. Sopin, Kazan Chemical Products Scientific Research Institutel [Abstract] Using ionizing radiation, which makes it possible to implement chemical transformations in the bulk of a polymer, is a popular method of modifying cellulose and cellulose-containing materials. The ionizing radiation used causes a series of changes in the physicochemical properties of the irradiated polymer that are determined by the ion-radical products of its radiolysis and the mechanism of their subsequent transformation. The study reported here used the electron spin resonance (EPR) method to study the kinetics of the accumulation of stable radicals in cellulose specimens irradiated at room temperature as a function of irradiation dose, energy of the electron beams, depth of the irradiated layer, and type of cellulose material. This was done to gather data for use in optimizing the process of the radiation chemical destruction of cellulose. Two types of air-dried cellulose were used: cotton pulp in the form of fiber (A) and sulfite cellulose in the form of paper 0.5 mm thick (B). The specimens were irradiated at room temperature. The EPR spectra were recorded on a BRUKER ER-200 radiospectrometer equipped with an ASPECT-2000 microcomputer. An ER-4105 DR

double resonator was used to determine the concentration of radicals. The EPR spectra obtained were compared with a standard containing a known quantity of paramagnetic centers, and the concentration integrals were determined by using a microcomputer. The error of the method ranged from 20-30% when determining absolute concentrations but did not exceed 5-10% during relative measurements. The EPR spectra of the two types of specimens were identical and were, according to literature data, the superimposition of a triplet with the component intensity ratio 1:2:1 and a split at 2.6 plus or minus 0.1 mT, a doublet with a split at 3.1 plus or minus 0.1 mT, and a singlet. The doublet and triplet signals observed were attributed to radicals occurring as a result of the breaking of the C-H bonds in the pyranose cycles of the cellulose at the 1 and 4 carbon atoms, respectively. The nature of the radicals with the singlet signal remained unclear. The kinetics of the accumulation of radicals in the cellulose specimens as a function of the irradiation dose were found to be well described by the equation $R = R_{lim}[1 - exp(-K_{deg}D)]$, where R_{lim} is the limiting (stationary) concentration of radicals and K_{deg} is the constant of their radiation-induced degradation. The radiation yield of radicals for the type B cellulose turned out to be noticeably lower than for the type A cellulose and increased slightly as the electron beam energy increased. Among other things, it was determined that when the unevenness of irradiation is less than 20%, the optimum number of layers for the type B specimens (regardless of irradiation dose) was n = 4 for electrons with an energy of 300 keV and n = 6 for electrons with an energy of 400 keV. Figures 3, table 1; references 6 (Russian).

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